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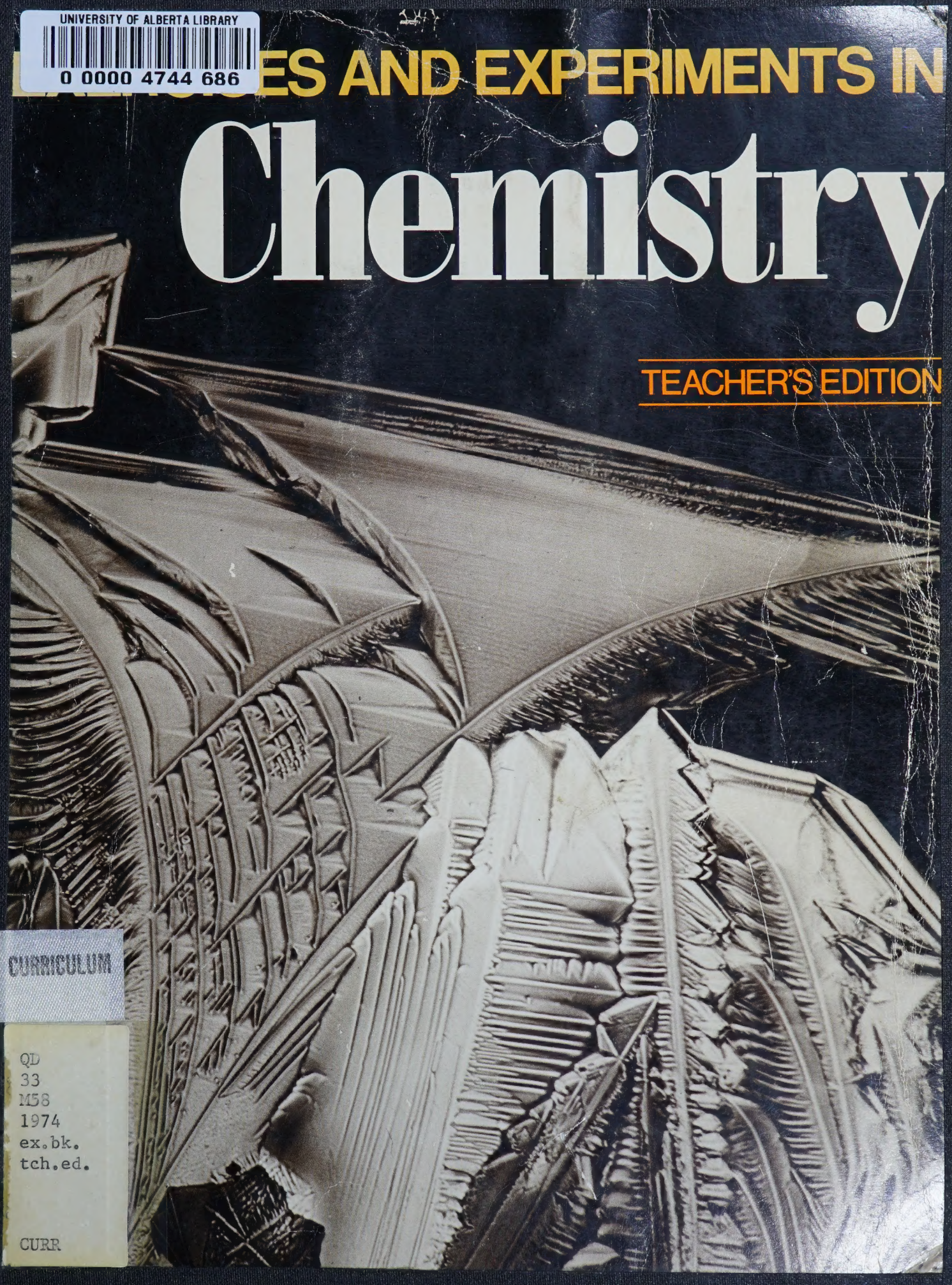

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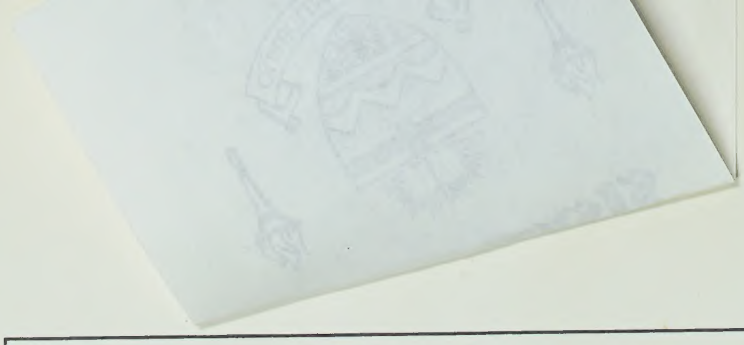

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Teacher's Edition

Exercises and Experiments in CHEMISTRY

H. Clark Metcalfe
John E. Williams
Joseph F. Castka

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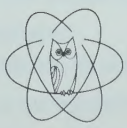

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Preface

The topics in *Exercises and Experiments in Chemistry* are arranged to follow the order of presentation in MODERN CHEMISTRY, 1974 edition. *Exercises and Experiment in Chemistry* may be used with any current high school chemistry textbook by adjusting the sequence of topics.

The exercise section is divided according to the unit organization of the 1974 edition of MODERN CHEMISTRY. The exercises supplement the work in the textbook. It is recommended that the assignment from the textbook be studied before the corresponding exercise is undertaken. For the most part the exercises follow the section sequence in MODERN CHEMISTRY except where the question format requires complete chapter coverage. The complete exercise serves as a good review before a scheduled test. The Review Unit exercises are especially useful for reviewing relatively large segments of subject matter. On occasion, the teacher may have students complete an exercise using the open textbook in class. Answers may be read during the last part of the period permitting students to check their own papers. The perforated pages permit teachers to collect exercises for checking. The authors recommend that the exercises be kept intact for teacher review. The variety of ways in which the exercises may be used helps to establish good work and study habits. Complete answers to the exercises appear in the *Teacher's Edition Exercises and Experiments in Chemistry*. On the completion of a chapter and/or unit, the class may be given the appropriate test from *Tests in Chemistry,* 1974.

The laboratory experiments are separated from the exercises and placed in the second part of *Exercises and Experiments in Chemistry*. These are the same experiments, numbered and arranged in the same sequence as those in *Laboratory Experiments in Chemistry*.

The format of the experiments in *Exercises and Experiments in Chemistry* provides blank spaces for observations, questions, equations, etc. Data tables are full-sized for actual entry of observations. Space for calculations is provided in many instances. Pages are perforated for tearing out so that completed experiments may be collected and checked by the teacher.

The 65 experiments represent an extensive and comprehensive list from which the teacher may select an appropriate number of experiments for the school year. The teacher should select those experiments which best fit the aims of the course, the needs of the students, and the time allotted for laboratory work. Time permitting, additional experiments may be used for enrichment and/or investigation by students who display particular aptitude or interest.

Certain experiments or selected portions may be used as teacher demonstrations, supplementing those which constitute the selected laboratory course. Many experiments include a section entitled *Further Experimentation* which may serve to stimulate and guide project activity. Each experiment contains, clear, workable directions which require apparatus and materials which are usually available.

The arrangement follows the sequence of the 1974 edition of MODERN CHEMISTRY in order to provide an experimental background for each chapter. Experiments should be performed before the formal class presentation to emphasize the experimental approach to the study of chemistry, when feasible. The *Introduction* to each experiment sets the stage for the experimental investigation of the problems stated under the section entitled *Purpose*. Many of the experiments are of the open-ended type in which the student cannot find the answer by referring to the textbook.

Most of the quantitative-theoretical experiments appear in the first half, since this paralleles the concentration of this type of material in the text. The experiments can roughly be divided into the following categories:

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1. Descriptive-observational, in which students observe, summarize, and draw conclusions.
2. Qualitative, in which the students record observations in data tables and draw conclusions on the basis of qualitative comparison.
3. Quantitative, in which students measure various quantities, and make computations based on quantitative observations.

The *Teacher's Edition Exercises and Experiments in Chemistry* contains lists of students apparatus and additional laboratory equipment, a list of needed chemicals, directions for preparation of solutions, and answers to the questions, observations, and Data Tables where feasible.

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Exercise 1

NAME . . . . . PERIOD . . . . . DATE . . . . . .

INSTRUCTOR'S COMMENTS . . . . . .

The Science of Chemistry

DIRECTIONS: In the parentheses at the right of each word or expression in the second column, write the letter of the expression in the first column which is most closely related.

a. logical approach to the solution of a problem	mathematics	(f)	1
b. measure of inertia	energy	(d)	2
c. measure of the earth's attraction	scientific method	(a)	3
d. capacity for doing work	inertia	(j)	4
e. mass per unit volume	matter	(k)	5
f. language of chemistry	weight	(c)	6
g. physical property	mass	(b)	7
h. definite volume, no definite shape	density	(e)	8
i. chemical property	liquid	(h)	9
j. resistance to change of position	boiling point	(g)	10
k. occupies space and has mass			
l. energy of motion			

DIRECTIONS: In the blank space at the right of each statement, write the word or expression which BEST completes the meaning when substituted for the corresponding number.

11. Since chemistry deals with the composition of materials and the changes in composition of materials, it is known as a(n) .(11). science. — physical 11

12. Observing, generalizing, theorizing, and testing are phases in the application of .(12). — scientific methods 12

13. A plausible explanation of an observed phenomenon in terms of a simple model which has familiar properties is called a(n) .(13). — theory 13

14. We may recognize the existence of .(14). through our senses. — matter 14

15. All matter offers resistance to change in position or motion due to its .(15). — inertia 15

16. When we measure this resistance of a body to any change in position or motion, we are measuring the .(16). of the body. — mass 16

17-18. In chemistry, we determine the .(17). of a body by weighing it on a chemical balance and sometimes incorrectly refer to this as the .(18). of the body. — mass . . . . . 17; weight 18

19. Matter which has both definite volume and shape is said to be in the .(19). phase. — solid 19

20. Vapor is the term used for the gaseous phase of a material which is a(n) .(20). under normal conditions. — liquid or solid 20

21. Liquids and gases are known as .(21). because they flow readily. — fluids 21

22. The properties of a material which can be determined without causing a change in the identity of the material are .(22). properties. — physical 22

23. The properties which describe the behavior of a material during a change in which its identity is altered are .(23). — chemical 23

24-25. Matter and energy are .(24)., and the total matter and energy in the universe is .(25). — interchangeable . . . 24; constant . . . 25

26. On the Celsius temperature scale the normal freezing point of water is .(26). degrees. — zero . . . 26

27. There are .(27). degrees of temperature difference between the freezing point and the normal boiling point of water on the Celsius scale. — 100 . . . 27

28. In an ordinary chemical change, the total mass of reacting materials is .(28). the total mass of products. — equal to . . . 28

29. The .(29). of a system is a measure of its ability to transfer heat to, or acquire heat from, other systems. — temperature . . . 29

30. The quantity of heat required to raise the temperature of 1 gram of water 1 Celsius degree is .(30).. — 1 calorie . . . 30

31. In measurement, the difference between the observed value and the accepted value is the .(31). — absolute error . . . 31

32. When measured quantities are multiplied or divided, the uncertainty in the result is the sum of the .(32). deviations of the individual measurements. — relative . . . 32

33. In the centigram balance, the typical tolerance shown as uncertainty is ± .(33). — 0.01 g . . . 33

34. A set of measurements includes the following individual values: 25.64 g, 25.58 g, 25.62 g, and 25.44 g. The "best" value for the average value of the set is .(34). — 25.57 g . . . 34

DIRECTIONS; Place the properly labeled answers to the following questions in the space provided at the right.

35. How many centimeters are there in 5.00 inches? — 12.7 cm . . . 35

36. What is the area of the bottom of a box measuring 10.0 cm long, 15.0 cm wide, and 0.255 m deep? — 150 cm^2 . . . 36

37. What is the volume, in liters, of the box in Question 36? — 3.82 liters . . . 37

38. A gas sample having a mass of 2.016 g occupies 22.4 liters. What is the density of the gas? — 0.0900 g/liter . . . 38

39. Express the sum of 25.0 cm, 225 mm, and 0.275 m in centimeters. — 75.0 cm . . . 39

40. What is the difference, in milligrams, between 1.24×10^{-3} g and 1.4×10^{-4} g? — 1.10 mg . . . 40

41. A measurement of 26.75 cm was recorded using a straight edge graduated in millimeters. How many significant figures are contained in the recorded measurement? — four . . . 41

42. Using the straight edge of Question 41, a student determined the dimensions of a box to be 10.65 cm long, 5.25 cm wide, and 4.25 cm deep. What is the volume of the box in cubic centimeters? — 238 cm^3 . . . 42

Exercise 2

NAME. . . . . PERIOD. . . . . DATE. . . . . .
INSTRUCTOR'S COMMENTS . . . . . .

Matter and its Changes

DIRECTIONS: Write the answers to the following in the space provided. Where appropriate, make complete statements.

1. What three general classes of matter do chemists recognize? . . . . . .

The three general classes of matter are (1) elements, (2) compounds, and (3) mixtures. 1

2. How can you recognize that granite is a mixture of different materials? . . . . . .

Close examination of a surface of a piece of granite reveals the presence of three distinctly different kinds of materials. . . . . .

. 2

3. What is a heterogeneous material? . . . . . .

A heterogeneous material is one made up of parts possessing different properties. 3

4. What is a homogeneous material? . . . . . .

A homogeneous material is one which has similar properties throughout. 4

5. What kind of mixture is not heterogeneous? . . . . . .

A solution is an example of a mixture that is not heterogeneous. 5

6. How can you tell that ordinary solutions are mixtures? . . . . . .

The amounts of materials making up solutions can be varied, and the characteristics of each material may be recognized. . . . . .

. 6

7. What is the definition of a mixture? . . . . . .

A mixture is a material consisting of two or more kinds of matter, each keeping its own characteristic properties. 7

8. What is an example of a homogeneous material that cannot be called a pure substance?

A solution is homogeneous but consists of a mixture of two or more substances. 8

9. Pure substances may be of what two kinds? . . . . . .

Pure substances may exist as compounds and elements. 9

10. What distinction is made between these two kinds of pure substances? . . . . . .

Compounds are complex substances which can be decomposed by chemical means into two or more simpler substances. . . . . .

elementary substances which cannot be further decomposed by chemical means. . . . . .

. 10

11. Write the symbols for (a) the two elements which together account for almost 75% of the continental crust, (b) the four elements whose compounds make up almost all of the earth's mantle, (c) the two metals which make up most of the earth's core. (a) O, Si. . . . . .

. . . . . ., (b) Mg, Fe, Si, O. . . . . ., (c) Fe, Ni. 11

12. What are the two general classes of elements? The two general classes of elements are: metals and nonmetals. Elements called metalloids have certain properties characteristic of metals and others of nonmetals. 12

13. What are the two general rules of capital letters that are used in writing the symbols of elements? . . . . . .

The first letter of a symbol is always capitalized. The second letter of a two-letter symbol is never capitalized. 13

14. What does a chemical symbol represent quantiatively? .....

A chemical symbol represents one atom of a certain element. 14

15. What is an atom? .....

An atom is the smallest unit of an element that can enter into combinations with other elements. 15

16. What is a statement of the law of definite composition .....

Every compound has a definite composition by weight. 16

17. In what sort of changes do the identifying properties of substances remain unchanged?

physical changes 17

18. Changes in which different substances with new properties are formed are called .....

chemical changes 18

19. Chemical changes involve either the absorption or liberation of energy. Briefly state another type of evidence which may show that a chemical change has taken place. .....The production of a gas is usually evidence that a chemical change is taking place. The formation of an insoluble solid, called a precipitate, may show that a chemical change has taken place as solutions are mixed. 19

20. List the five means by which chemists bring about chemical changes or control those which have already started. .....

Heat energy, light energy, electric energy, solution in water, catalysis. .....

.....20

21. That property which describes the disorder of a system is called .....

entropy 21

22-23. Processes in nature are driven in two ways: toward .....lowest energy, ...22

and toward .....highest entropy 23

24. In a nuclear change the new substance(s) is (are) formed by changes in the .....

identity of the atoms themselves. 24

25. Directions: Fill in the spaces in the table below with either a *Yes* or *No* answer to indicate the general properties of metals and nonmetals.

	Metals	Nonmetals
a. usually have a luster	Yes	No
b. are often malleable	Yes	No
c. may be gaseous at room temperature	No	Yes
d. are generally poor conductors of heat	No	Yes
e. are usually good conductors of electricity	Yes	No

DIRECTIONS: Various changes in matter are listed below. Indicate by letter which are characteristically physical P, which are chemical C, and which are nuclear N.

26. iron rusts	C	31. coal burns	C
27. ice melts	P	32. alcohol evaporates	P
28. radium decays to lead	N	33. a rocket is launched	C
29. sugar dissolves in water	P	34. steam condenses	P
30. milk sours	C	35. limestone is crushed	P

NAME . . . . . PERIOD . . . . . DATE . . . . .
INSTRUCTOR'S COMMENTS . . . . . .

Exercise 3 Atomic Structure

DIRECTIONS: Complete the following statements, forming accurate and complete sentences.

1. Dalton was the first to realize that the two observations about matter which suggested the nature and properties of atoms were (a) the law of definite composition (b) the way and proportions in which substances react with one another. 1

2. All matter is made up of very small particles called atoms. 2

3. Atoms of the same element are chemically alike; atoms of different elements are chemically different 3

4. While individual atoms of a given element may not all have the same mass, the atoms of an element, as it occurs naturally have, for practical purposes, a definite average mass characteristic of the element. 4

5. While individual atoms of different elements may have nearly identical masses, the atoms of different naturally occurring elements have different average masses. 5

6. Atoms are not subdivided during chemical reactions. 6

7. The central part of an atom is called (a) the nucleus, is charged (b) positively, is very dense and very small, and has a diameter which is about (c) 10^{-4} Å. 7

8. In Rutherford's experiment, the few slightly deflected and the very few greatly deflected high speed positively charged particles were explained by assuming that the particles were turned from their path by the nucleus 8

9. Electrons moving about the nucleus and having similar energies comprise a(n) (a) shell and are said to be in the same (b) energy level 9

10. Each atom is electrically neutral, since the total positive charge of the nucleus is equal to the total negative charge of the electrons in the shells. 10

DIRECTIONS: Fill in the blank spaces below with information about each of the following:

	Mass in grams	Mass number	Charge	Position in the atom
11. electron	9.110×10^{-28} g	0	−1	in the shells 11
12. proton	1.673×10^{-24} g	1	+1	in the nucleus 12
13. neutron	1.675×10^{-24} g	1	0	in the nucleus 13

DIRECTIONS: Write answers to the following in the spaces provided. Where appropriate, make complete statements.

14. Scientists discover how electrons are arranged in atoms of an element by studying the chemical properties of the element and its spectrum.

15. The atomic number of an atom is the number of protons in the nucleus 15

16. Isotopes are atoms of the same element which have different masses. 16

17. Isotopes are nuclides having the same atomic number. 17

18. The mass number of an atom is the sum of the number of protons and neutrons in its nucleus. 18

19. A certain atom consists of 16 protons, 18 neutrons, and 16 electrons. What is its (a) atomic number? 16 (b) mass number? 34 (c) distribution of electrons in shells? two K-shell electrons, eight L-shell electrons, six M-shell electrons .19

20. What is the atomic mass of an atom?

The mass of an atom expressed relative to the carbon–12 = exactly 12 scale. .20

21. What is the relationship between mass number and atomic mass of an atom?

The mass number is the whole number closest to the atomic mass. .21

22. Why is the Avogadro number said to be a constant? The Avogadro number is a constant because the number of atoms in the gram-atomic mass of any nuclide is the same, 6.022169×10^{23} atoms. .22

23. To three significant figures state the value of the Avogadro number. 6.02×10^{23} .23

24. What is a mole of a substance?

A mole of a substance is the amount of the substance containing the Avogadro number of any kind of chemical unit. .24

25. What is the gram-atomic weight of an element?

The gram-atomic weight of an element is the mass in grams of one mole of naturally occurring atoms of an element. .25

26. How is the atomic weight of an element related to its gram-atomic weight?

The numerical portion of the gram-atomic weight of an element is its atomic weight. .26

27. The atomic weight of fluorine is 18.9984. (a) How many atoms are there in 4.7496 g of this element? 1.5055×10^{23} atoms. (b) How many moles of atoms? 0.25000 mole. .27

28. The mass of 3.01108×10^{23} atoms of nitrogen-14 is 7.00154 g. What is the atomic mass of this nuclide? 14.0031 .28

29. What is the mass in grams of 2.50 moles of gallium atoms? 174 g .29

30. How many moles of atoms are there in 8.994 g of aluminum? 0.3333 mole .30

31. Complete the following table:

Name of nuclide	Atomic number	Mass number	Composition of nucleus		Electron configuration		
			Protons	Neutrons	K	L	M
hydrogen-2 (deuterium)	1	2	1	1	1		
lithium-7	3	7	3	4	2	1	
nitrogen-14	7	14	7	7	2	5	
fluorine-19	9	19	9	10	2	7	
magnesium-24	12	24	12	12	2	8	2
silicon-28	14	28	14	14	2	8	4
sulfur-32	16	32	16	16	2	8	6
argon-40	18	40	18	22	2	8	8

Exercise 4

NAME . . . . . PERIOD . . . . . DATE . . . . . .
INSTRUCTOR'S COMMENTS . . . . . .

Arrangement of Electrons in Atoms

DIRECTIONS: Complete the following statements, forming accurate and complete sentences.

1. The nucleus has a positive charge because of its protons. A neutral atom contains an equal number of protons and of negatively charged electrons. Therefore, we might expect electrons to be held in an atom by the . attraction between oppositely charged particles. 1

2. The observed behavior of oppositely charged particles is not satisfactory for explaining the motion of electrons about the nucleus of an atom because we know that atoms do not collapse. 2

3. The speed of any wave motion equals the product of . . . . . The frequency and the wavelength. 3

4. The energy of a photon, E, may be represented by the expression, $E =$. hf 4

5. When excited atoms radiate energy, the radiation is given off in units called. photons 5

6. When excited atoms return to their normal states and the light they emit is passed through a spectroscope, we may observe a(n) . bright-line spectrum. 6

7. Energy changes within an atom do not provide a continuous flow of energy, but are fixed, definite amounts of energy. 7

8. The kinds of spectra which are explained satisfactorily by the Bohr model of the atom are . the spectra of one-electron particles. 8

9. A space orbital is . a highly probable location in which an electron may be found. 9

10. The interpenetration of one free atom by another is prevented by . the electron cloud. 10

11. The quantum number which indicates the average distance of the electron from the nucleus of the atom is the . principal quantum number. 11

12. The number of orbital shapes possible in the 2nd energy level is . two 12

DIRECTIONS: 13-16. For each sublevel type, furnish the required information.

Sub-level	Number of space orbitals	Maximum number of electrons	Lowest energy level having this sublevel type	
s	1	2	1st	13
p	3	6	2nd	14
d	5	10	3rd	15
f	7	14	4th	16

DIRECTIONS: Write answers to the following in the spaces provided. Make complete statements where appropriate.

17. What is the most stable state of an atom called? The most stable state of an atom is the ground state. 17

18. Under what conditions may two electrons occupy the same space orbital? .....

Two electrons may occupy the same orbital if they have opposite spins. 18

19. What is an electron pair? .....

An electron pair consists of two electrons of opposite spin in the same space orbital. 19

20. What name is given to an outer shell containing eight electrons? An outer shell containing eight electrons is called an octet. 20

21. (a) Which sublevels of the 3rd energy level are filled in the element argon (Ar-atomic number 18)? 3*s* and 3*p* are filled in the element argon. .....

(b) In the element krypton (Kr-atomic number 36)? The 3*s*, 3*p*, and 3*d* are filled in the element krypton. 21

22. When do successive electrons entering the 2*p* orbitals start to pair up? .....

Electrons do not pair up in the 2*p* orbitals until each of the three 2*p* space orbitals is occupied by a single electron. 22

23. What is the electron-dot symbol for potassium (atomic number 19)? K· 23

24. In what order do successive electrons usually occupy the sublevels of the atoms of the fourth series? Successive electrons usually occupy the orbitals of the fourth series elements in the order 4*s*, 3*d*, 4*p*. 24

25. Which atoms in the fourth series have structures which appear to be irregular because of the stability of (a) a half-filled 3*d* sublevel? chromium, ...

(b) a completely filled 3*d* sublevel? copper. 25

26. Write the electron-dot symbol for an atom having an atomic number of 13 and a mass number of 27 .. Al: 26

27. In which series of elements do electrons occupy the 4*f* sublevel? .....

Electrons occupy the 4*f* sublevel in the sixth series. 27

28. Complete the following table:

Symbol-Atomic number	Electron configuration notation	Electron-dot notation	Orbital notation 1*s*	2*s*	2*p*	3*s*	3*p*	3*d*	4*s*
Li-3	$1s^2 2s^1$	Li·	⊗	⊘					
B-5	$1s^2 2s^2 2p^1$	·B:	⊗	⊗	⊘ ○ ○				
N-7	$1s^2 2s^2 2p^3$	·N:	⊗	⊗	⊘ ⊘ ⊘				
F-9	$1s^2 2s^2 2p^5$	:F:	⊗	⊗	⊗ ⊗ ⊘				
Mg-12	$1s^2 2s^2 2p^6 3s^2$	Mg:	⊗	⊗	⊗ ⊗ ⊗	⊗			
Si-14	$1s^2 2s^2 2p^6 3s^2 3p^2$	·Si:	⊗	⊗	⊗ ⊗ ⊗	⊗	⊘ ⊘ ○		
S-16	$1s^2 2s^2 2p^6 3s^2 3p^4$	·S:	⊗	⊗	⊗ ⊗ ⊗	⊗	⊗ ⊘ ⊘		
Ar-18	$1s^2 2s^2 2p^6 3s^2 3p^6$	:Ar:	⊗	⊗	⊗ ⊗ ⊗	⊗	⊗ ⊗ ⊗		
Ca-20	$1s^2 2s^2 2p^6 3s^2 3p^6 4s^2$	Ca:	⊗	⊗	⊗ ⊗ ⊗	⊗	⊗ ⊗ ⊗	○ ○ ○ ○ ○	⊗
Sc-21	$1s^2 2s^2 2p^6 3s^2 3p^6 3d^1 4s^2$		⊗	⊗	⊗ ⊗ ⊗	⊗	⊗ ⊗ ⊗	⊘ ○ ○ ○ ○	⊗

Exercise 5

NAME . . . . . PERIOD . . . . . DATE . . . . . .

INSTRUCTOR'S COMMENTS . . . . . .

The Periodic Law

DIRECTIONS: Write answers to the following in the spaces provided. Where appropriate, make complete statements.

1. Who did the pioneer work on the Periodic Table we use today? . . . . . Mendeleyev. 1

2. What important use did he make of his Periodic Table? . . . . . .

He predicted that elements would be discovered to fill the gaps he left in his Periodic Table, and he predicted the properties these elements would have. 2

3. In what order are the elements listed in our present Periodic Table? . . . . . .

They are listed in order of increasing atomic numbers. 3

4. How did Moseley account for the fact that the X-ray wavelengths obtained from two successive known elements sometimes had twice the expected variation? . . . . . .

An undiscovered element belonged between them in the Periodic Table. 4

5. State the periodic law. . . . . .

The physical and chemical properties of elements are periodic functions of their atomic numbers. 5

6. What name is given to the elements in a vertical column of the Periodic Table? . . . . . .

Group or family. 6

7. What name is given to the elements in a horizontal row of the Periodic Table? . . . . . .

Period or series. 7

8. What name is given to elements whose atoms usually differ in electron configuration by the entrance of successive electrons in a *d* sublevel? . . . . . .

Transition elements. 8

9. What name is given to elements whose atoms usually differ in electron configuration by the entrance of successive electrons in an *f* sublevel? . . . . . .

Rare earth elements. 9

10. Fill in the blocks with the symbols, atomic numbers, electron-configuration notation, and brief statements of properties of the elements of the second and third periods as has been done for beryllium and magnesium below.

Li 3, Soft, silvery, active metal	$1s^2$ $2s^1$	Be 4 silvery metal	$1s^2$ $2s^2$	B 5, Black solid, few metallic properties	$1s^2$ $2s^2$ $2p^1$	C 6, Solid, properties between metals and nonmetals	$1s^2$ $2s^2$ $2p^2$	N 7, Colorless gas, nonmetallic properties	$1s^2$ $2s^2$ $2p^3$	O 8, Colorless gas, strong nonmetallic properties	$1s^2$ $2s^2$ $2p^4$	F 9, Pale-yellow gas, very strong nonmetallic properties	$1s^2$ $2s^2$ $2p^5$	Ne 10, Colorless, inert gas	$1s^2$ $2s^2$ $2p^6$
Na 11, Soft, silvery, active metal	$1s^2$ $2s^2$ $2p^6$ $3s^1$	Mg 12 silvery metal	$1s^2$ $2s^2$ $2p^6$ $3s^2$	Al 13, Silvery metal, some nonmetallic properties	$1s^2$ $2s^2$ $2p^6$ $3s^2$ $3p^1$	Si 14, Dark nonmetallic element	$1s^2$ $2s^2$ $2p^6$ $3s^2$ $3p^2$	P 15, Solid nonmetallic element	$1s^2$ $2s^2$ $2p^6$ $3s^2$ $3p^3$	S 16, Solid, yellow, nonmetallic	$1s^2$ $2s^2$ $2p^6$ $3s^2$ $3p^4$	Cl 17, Yellow-green gas, strong nonmetallic properties	$1s^2$ $2s^2$ $2p^6$ $3s^2$ $3p^5$	Ar 18 Colorless inert gas	$1s^2$ $2s^2$ $2p^6$ $3s^2$ $3p^6$

11. Fill in the blocks with the names, symbols, atomic numbers, electron configurations, and other designated information.

Group I	Group II		Group V		Group VII		Group VIII	
							helium He 2	2
lithium Li 3	2 1	beryllium Be 4	2 2	nitrogen N 7	2 5	fluorine F 9	2 7	neon Ne 10	2 8
sodium Na 11	2 8 1	magnesium Mg 12	2 8 2	phosphorus P 15	2 8 5	chlorine Cl 17	2 8 7	argon Ar 18	2 8 8
potassium K 19	2 8 8 1	calcium Ca 20	2 8 8 2	arsenic As 33	2 8 18 5	bromine Br 35	2 8 18 7	krypton Kr 36	2 8 18 8
rubidium Rb 37	2 8 18 8 1	strontium Sr 38	2 8 18 8 2	antimony Sb 51	2 8 18 18 5	iodine I 53	2 8 18 18 7	xenon Xe 54	2 8 18 18 8
cesium Cs 55	2 8 18 18 8 1	barium Ba 56	2 8 18 18 8 2	bismuth Bi 83	2 8 18 32 18 5	astatine At 85	2 8 18 32 18 7	radon Rn 86	2 8 18 32 18 8
francium Fr 87	2 8 18 32 18 8 1	radium Ra 88	2 8 18 32 18 8 2						

Name of family	Sodium Family	Calcium Family	Nitrogen Family	Halogen Family	Noble Gas Family
Nature of properties	Very active metallic	Active metallic	Nonmetallic to metallic	Very active nonmetallic	Top 3 inert bottom 3 reactive
Direction of increasing activity	Generally from top to bottom of table	From top to bottom of table		From bottom to top of table	

12. What arrangement of outer-shell electrons identifies elements with generally similar properties? . . Similar arrangement of outer-shell electrons identifies elements with generally similar properties. 12

13. Excluding the noble gases, state the location in the periodic table of (a) the most active metal, and (b) the most active nonmetal. (a) lower left corner (b) upper right corner .13

14. Which of the noble gases have no known compounds? . . . . . .

No compounds of helium, neon, and argon are known. 14

15. What does the electron population of the outermost shell of the atoms of the transition elements reveal about their properties? . . . The transition elements are metals. 15

16. Why do elements of the lanthanide series have almost identical chemical properties?

The two outer shells of the atoms of these elements are almost the same. 16

17. What is the significance of the zigzag line running diagonally down and to the right near the right end of the periodic table? . . . This zigzag line separates the metals from the nonmetals. 17

18. What properties do metalloids show? . . . Metalloids show both metallic and nonmetallic properties under different conditions. 18

19. Why isn't the volume of an atom a completely definite quantity? . . . The volume of an atom is not a completely definite quantity because the boundary of an atom's electron cloud is not a distinct surface, but is somewhat fuzzy and indefinite. 19

20. Compose two statements describing the periodicity of atomic radii. . . . . .

(a) In a family of elements, the atomic radius increases with atomic number. . . . . .

(b) In a period of elements, the atomic radius generally decreases from Group I to Group VIII. 20

21. In the reaction, A + energy → $A^+ + e^-$, (a) what kind of a particle is A^+? . . . . . .

A singly-charged positive ion. . . . . .

(b) What name describes the energy involved? . . . Ionization energy. 21

22. In Question 21, what kind of element is A generally if the quantity of energy (a) is low? A metal. . . . . (b) is high? . . . A nonmetal. . . . (c) is of intermediate value? . . . A metalloid. 22

23. What two properties of the noble gases furnish strong evidence for the unusual stability of the outer-shell octet? . . . The chemical inertness and the unusually high ionization energies. 23

24. (a) How does the ionization energy vary with atomic number within a group? (b) Why?

(a) Ionization energy generally decreases with increasing atomic number. (b) . . . The outer-shell electrons of the elements of higher atomic number are farther from the nucleus causing increasing atomic radius. 24

25. Explain the decrease in first ionization energy between Groups II and III in Periods 2 and 3. . . . The *s* sublevel is filled and the *p* sublevel is started. 25

26. Between which two other groups in Periods 2 and 3 is there another decrease in first ionization energy? . . . There is a decrease between Group V and Group VI as the *p* sublevel becomes half-filled. 26

27. Explain the sharp decrease in ionization energy between the last transition element and the Group III element in Period 4. . . . The *d* sublevel has become filled and the *p* sublevel is started. 27

28. What apparently causes the observed irregularities in the general increase in ionization energy in each series? . . . The irregularities are apparently caused by the extra stability of completed and half-completed sublevels. 28

29. Why is the ionization energy required to remove the second electron from Na atoms so very much greater than that needed to remove the first electron? . . . Each successive electron must be removed from a particle with increasingly greater net positive charge. The second electron is a 2*p* electron. 29

30. Why is the ionization energy needed to remove the first two electrons from Mg atoms relatively low? .....

Both electrons are 3*s* electrons. 30

31. Why is it easier to remove the first electron from Al atoms than it is to remove the first electron from Mg atoms? .....

The first aluminum electron is a 3*p* electron which is in a slightly higher energy sublevel than the first magnesium electron which is a 3*s* electron. 31

32. In the reaction, $A + e^- \rightarrow A^- +$ energy, (a) what kind of a particle is A^-? ..... (b) what name describes the energy involved? (a) A singly charged negative ion. (b) Electron affinity. 32

33. What are the present uses of the Periodic Table? .....

It is a useful, systematic classification of elements according to their properties which helps make the study of chemistry easier. 33

Exercise 6

NAME . . . . . PERIOD . . . . . DATE . . . . . .

INSTRUCTOR'S COMMENTS . . . . . .

Chemical Bonds

DIRECTIONS: In the blank space at the right of each statement, write the word or expression which BEST completes the meaning when substituted for the corresponding number.

1-2. The electrons in an incomplete outer shell of an atom are called the .(1). electrons. The balance of the atom is called the .(2).

valence 1

kernel 2

3. The transfer or sharing of valence electrons between atoms produces chemical .(3). between the atoms.

bonds 3

4. Electron transfer results in .(4). bonding.

ionic 4

5. Electron sharing produces .(5). bonding.

covalent 5

6-7. When one atom combines with another atom, both atoms may attain an outer shell consisting of a(n) producing a structure that has .(7).

octet 6

chemical stability 7

8-9. The process of spontaneous electron transfer always involves the .(8). of energy. If energy is absorbed during electron sharing, the process is said to be .(9).

release 8

endothermic 9

10. A shorthand method of using chemical symbols to represent the composition of a compound is known as a(n) .(10).

chemical formula 10

11-12. A sodium ion has a single excess .(11). charge and a chloride ion has a single excess .(12). charge.

positive 11

negative 12

13. The electron-dot symbol for the sodium atom is .(13).

$Na\cdot$ 13

14. The electron-dot symbol for the chlorine atom is .(14).

$:\ddot{Cl}:$ 14

15. In the electron-dot formula for sodium chloride the chloride ion is represented as .(15).

$:\ddot{Cl}:$ 15

16. The amount of energy required to remove one mole of electrons from one mole of sodium atoms is the .(16).

ionization energy 16

17. The amount of energy released when one mole of chloride ions is produced from one mole of chlorine atoms is the .(17).

electron affinity 17

18. The energy change in the formation of all ionic compounds from their elements is .(18).

exothermic 18

19. Any chemical reaction which involves the loss of one or more electrons by an atom or ion is a(n) .(19).

oxidation 19

20. The oxidation number of chlorine in Cl_2 is .(20).

zero 20

21. The substance which is reduced during a chemical change is called the .(14). agent.

oxidizing 21

22. The algebraic sum of the oxidation numbers of all the atoms in the formula of a compound is .(22).

zero 22

23-25. In the formation of magnesium bromide, the electron(s) transferred from the magnesium atom has (have) the electron-configuration notation .(23). The number of bromine atoms needed to react with one magnesium atom is .(24). The ionic formula for magnesium bromide is .(25).

$3s^2$ 23

two 24

$Mg^{++}\ Br_2^-$ 25

26. All formulas for ionic compounds are .(26). formulas.

empirical 26

27-28. Some .(27). elements can transfer the electrons in the outermost sublevel and sometimes with very little additional energy can transfer one or two electrons from the .(28). sublevel.

transition 27

next-to-outermost 28

29. The electron-dot formula for the ionic compound potassium bromide is .(29).

$K^+ \left[\cdot\underset{\cdot\cdot}{\overset{\cdot\cdot}{\mathrm{Br}}}\cdot\right]^-$ 29

30-31. Metallic ions are (larger, smaller) .(30). than the corresponding metallic atoms and nonmetallic ions are (larger, smaller) .(31). than the corresponding nonmetallic atoms.

smaller 30

larger 31

32-33. Positive ions are called .(32). while negative ions are called .(33).

cations 32

anions 33

34-35. Within a group of elements the ion size (increases, decreases) .(34). with atomic number because of .(35). addition.

increases 34

shell 35

36. Within a period of elements, the Group I, II, and III cations show a sharp .(36). in size.

decrease 36

37-39. The electron-dot formula for the diatomic hydrogen molecule is .(37). Its structural formula is .(38). Its shape is .(39).

$H\cdot H$ 37

$H-H$ 38

linear (straight-line) 39

40. The energy required to break chemical bonds is called the .(40).

bond energy 40

41-42. Both atoms of chlorine in a chlorine molecule have the stable electron arrangement of the noble gas .(41). with an octet in the .(42). energy level.

argon 41

third 42

43-44. The structure of a nitrogen molecule indicates the sharing of .(44). pair(s) of electrons. Its structural formula is .(44).

three 43

$N\equiv N$ 44

45. Each atom in the molecule of elements having diatomic molecules is assigned an oxidation number of .(45).

zero 45

46. The reaction for the formation of one mole of hydrogen chloride molecules from one-half mole of hydrogen molecules and one-half mole of chlorine molecules is a(n) .(46). reaction.

exothermic 46

47-48. The two possible shapes of a molecule composed of three atoms are described as being either .(47). or .(48).

linear 47

bent 48

49. Water molecules break up according to the equation: 1 mole H_2O + 222 kcal → 2 moles H + 1 mole O. The H—O bond energy is .(49). kcal/mole.

111 49

50. In compounds, the oxidation number of oxygen is usually .(50).

−2 50

51. An ammonia molecule has the shape of a(n) .(51).

pyramid 51

52. Methane molecules have a(n) .(52). shape.

tetrahedral 52

53. The concept of .(53). is an attempt to make up for the deficiencies of the electron-dot structures of certain molecules written in accord with the electron-pair and electron-octet rules.

resonance 53

54. The neutral particle which results from the covalent bonding of atoms is a(n) .(54).

molecule 54

55. The combining of two or more orbitals of nearly the same energy into new orbitals of equal energy is called .(54).

hybridization 55

56. The four equivalent orbitals of carbon in the methane molecule are called the .(55). orbitals.

Sp^3 56

57. The angles between the N--H bonds in ammonia are known to be 108°, which agrees quite well the .(56). angle value of 109.5°.

tetrahedral 57

58-59. A molecule of oxygen is represented by the formula .(58). while two separate unbonded oxygen atoms are written as .(59).

O_2 58

2O 59

60. A(n) .(60). of a symbol or formula gives the number of particles whose composition is given by the symbol or formula.

coefficient 60

61. A covalent bond in which there is an unequal attraction for the shared electrons and a resulting unbalanced distribution of charge is called a(n) .(34). covalent bond.

polar 61

62. Metals are characterized by (high, low) .(62). electronegativity.

low 62

63. Within each numbered group, electronegativity generally (decreases, increases) .(63). with increasing atomic number .

decreases 63

64. The lower the electronegativity, the (more, less) .(64). active is the metal.

more 64

65. In combinations involving nonmetals, the oxidation number of the less electronegative element is .(65)..

positive 65

66. If all the bonds in a molecule are nonpolar, the molecule is .(66).

nonpolar 66

67. If the polar bonds in a molecule are all alike, the polarity of the molecule as a whole depends only on the .(67). of the bonds.

spatial arrangement 67

68-69. Carbon dioxide and water have triatomic molecules in which the bonds are .(68). The carbon dioxide molecule is nonpolar since its shape is .(69).

polar 68

linear 69

70. Charged groups of covalently bonded atoms are called .(70). polyatomic ions 70

71. For the following expressions, write the oxidation number of the element whose symbol is underlined:

(a) $\underline{Fe}_2O_3$ ..... $^{+3}$ ..... (b) $K^+\underline{Mn}O_4^-$ ..... $^{+7}$ ..... (c) $\underline{P}O_4^{---}$ ..... $^{+5}$ .....

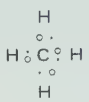
(d) $\underline{O}_2$ ..... ^{0} ..... (e) $H\underline{Cl}O_3$ ..... $^{+5}$ ..... (f) $\underline{N}H_4^+$ ..... $^{-3}$ .....

72. Draw the orbital notations for (a) F_2; (b) KCl

73. Draw electron-dot formulas for the following:

(a) H_2O

(c) CH_4

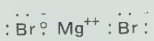


(e) CS_2

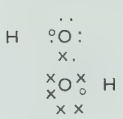
(g) HBr

(b) NH_3

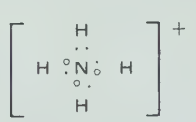
(d) $Mg^{++}Br_2^-$



(f) H_2O_2



(h) NH_4^+



Exercise 7

NAME ..... PERIOD ..... DATE .....

INSTRUCTOR'S COMMENTS .....

Chemical Composition

DIRECTIONS: In the blank space at the right of each statement, write the word or expression which BEST completes the meaning when substituted for the corresponding number.

1-2. The names of many ionic compounds are formed by giving in sequence the names of the .(1). of which they are composed. Where metals have more than one oxidation number, the name includes the oxidation number in .(2). in parentheses.

ions 1

Roman numerals 2

3-5. According to the ion-charge method for writing formulas, the total charge of the first (positive) part of the compound must be .(3). and opposite to the total charge of the second (negative) part of the compound. The total charge of an ion is found by multiplying the charge of the ion by the number of .(4). This method yields only a(n) .(5). formula.

equal 3

that ion taken 4

empirical 5

6-7. When a polyatomic ion is to be taken more than once in a formula, the polyatomic ion must be enclosed in .(6). and the number of times the polyatomic ion is taken represented by a(n) .(7). placed outside.

parentheses 6

subscript 7

8. Compounds consisting of two elements only are called .(8). compounds.

binary 8

9. The correct name for As_2S_5 is .(9).

diarsenic pentasulfide 9

10. When it is known that a substance exists as simple molecules, its formula is known as a(n) .(10). formula.

molecular 10

11. The formula representing the simplest whole-number ratio of atoms of the elements in a compound is the .(11). formula.

empirical 11

12. The formula weight of any compound is the .(12). of the atomic weights of all the atoms present in the formula.

sum 12

13. The formula weight of NaCl is .(13).

58.5 13

14. The term .(14). is a more general term than molecular weight and therefore is preferred by chemists.

formula weight 14

15. Atomic weights and formula weights are practically useful because that tell us the .(15). weights of elements or compounds that combine or react.

relative 15

16. The law of .(16). states that the percentage composition of a chemical compound is always the same regardless of the source.

definite composition 16

17. John Dalton recognized the possibility that two kinds of atoms could combine in more than one way and in more than one .(17).

proportion 17

18. The number of carbon-12 atoms in exactly 12 grams of this nuclide is approximately .(18).

6.02×10^{23} 18

19. The numerical portion of the gram-atomic weight of an element is the .(19). of the element.

atomic weight 19

20. The mass in grams of one mole of naturally-occurring atoms of an element is the .(20). of the element. gram-atomic weight 20

21. The number of grams of sulfur containing the same number of atoms as 5.00 moles of oxygen atoms is .(21). 160 g 21

22. The gram-molecular weight of a diatomic element is the mass of .(22). of molecules of the element. one mole 22

23. Since 1.00 mole of Cl atoms has a mass of 35.5 g, 1.00 mole of Cl_2 molecules has a mass of .(23). grams. 71.0 g 23

24. The mass of 2.00 moles of sodium chloride is .(24). grams. 117 24

25. The formula for a compound may represent the composition of the compound and the mass of .(25). of that compound. one mole 25

DIRECTIONS: In the space at the right, give the formulas for the following compounds.

26. calcium fluoride ..... CaF_2 .....
27. mercury(II) chloride ..... $HgCl_2$ .....
28. potassium nitrite ..... KNO_2 .....
29. antimony trichloride ..... $SbCl_3$ .....
30. barium carbonate ..... $BaCO_3$ .....
31. aluminum hydroxide ..... $Al(OH)_3$ .....
32. carbon monoxide ..... CO .....
33. silver chromate ..... Ag_2CrO_4 .....
34. iron(II) carbonate ..... $FeCO_3$ .....
35. copper(I) sulfide ..... Cu_2S .....
36. diarsenic pentasulfide ..... As_2S_5 .....
37. lead(II) phosphate ..... $Pb_3(PO_4)_2$ .....
38. ammonium nitrate ..... NH_4NO_3 .....
39. calcium hydroxide ..... $Ca(OH)_2$ .....
40. chromium(III) sulfite ..... $Cr_2(SO_3)_3$ .....
41. iron(II) oxide ..... FeO .....
42. magnesium acetate ..... $Mg(C_2H_3O_2)_2$ .....
43. sodium peroxide ..... Na_2O_2 .....
44. potassium chlorate ..... $KClO_3$ .....
45. zinc iodide ..... ZnI_2 .....

DIRECTIONS: Using approximate atomic weights of the elements, write the formula weight of each compound in the space provided at right.

46. KNO_3 ..... 101.1 .....
47. $NaOH$ ..... 40.0 .....
48. $CaCO_3$ ..... 100.1 .....
49. $PbSO_4$ ..... 303.3 .....
50. $CaSO_4$ ..... 136.2 .....
51. $(NH_4)_3PO_4$ ..... 149.0 .....
52. $AgC_2H_3O_2$ ..... 166.9 .....
53. $Cu(NO_3)_2$ ..... 187.5 .....
54. $Fe_3(PO_4)_2$ ..... 357.4 .....
55. $Na_2CO_3 \cdot 10H_2O$ ..... 286.0 .....

56-58. State the steps used to determine the percentage composition of a constituent element in a compound of known formula.

Step 1: Determine the formula weight by adding the atomic weights of all the atoms present. 56

Step 2: Express the total atomic weight contributed by the element of interest, the at. wt. X no. atoms, as a fractional part of the formula weight. 57

Step 3: Multiply this fractional part of the formula weight by 100% to give the percentage of the element present in the compound. 58

59-68. What is the percentage composition of each element present in each of the following compounds? Show each step in smooth form in the appropriate column.

Compound	Step 1	Steps 2 and 3	Percentage Composition
NaCl	58.5		
59. % Na	23.0	23.0 ÷ 58.5 × 100%	39.3% 59
60. % Cl	35.5	35.5 ÷ 58.5 × 100%	60.7% 60
K_2CO_3	138.2		
61. % K	78.2	78.2 ÷ 138.2 × 100%	56.6% 61
62. % C	12.0	12.0 ÷ 138.2 × 100%	8.7% 62
63. % O	48.0	48.0 ÷ 138.2 × 100%	34.7% 63
Fe_3O_4	231.4		
64. % Fe	167.4	167.4 ÷ 231.4 × 100%	72.3% 64
65. % O	64.0	64.0 ÷ 231.4 × 100%	27.7% 65
$C_3H_5(OH)_3$	92.0		
66. % C	36.0	36.0 ÷ 92.0 × 100%	39.1% 66
67. % H	8.0	8.0 ÷ 92.0 × 100%	8.7% 67
68. % O	48.0	48.0 ÷ 92.0 × 100%	52.2% 68

69-71. By what three steps can you determine the empirical formula of a compound if either the mass or mass percentage of each constituent element is known?

Step 1: Determine the relative number of moles of atoms of each element by dividing the mass of each element by the mass per mole of that element. (Percentage composition data is converted to relative mass data on the basis of a 100-gram sample) 69

Step 2: Find the smallest ratio of atoms by dividing the relative number of moles of atoms of each element by the smallest relative number of moles of atoms. 70

Step 3: Find the simplest whole-number ratio (if not found in step 2) by expressing as fractions and clearing. 71

72-80. What is the empirical formula of each compound for which the analysis is given below?

72. Analysis: H = 1.24%, Br = 98.76%. (see following page) ..... HBr 72

73. Analysis: K = 44.9%, S = 18.4%, O = 36.7%. ..... K_2SO_4 73

74. Analysis: Iron = 17.21 g, chlorine = 32.79 g. ..... $FeCl_3$ 74

75. Analysis: K = 28.71%, H = 0.73%, S = 23.57%, O = 46.99%. ..... $KHSO_4$ 75

76. Analysis: Ba = 80.2%, O = 18.7%, H = 1.2%. ..... $Ba(OH)_2$ 76

77. Analysis: Mg = 27.73%, P = 23.58%, O = 48.69%. ..... $Mg_3(PO_4)_2$ 77

78. Analysis: Cu = 39.79%, S = 20.11%, O = 40.10%. ..... $CuSO_4$ 78

79. Analysis: Mg = 20.18%, S = 26.66%, O = 53.16%. ..... $MgSO_4$ 79

80. A certain hydrocarbon was found to be 80.0% carbon and 20.0% hydrogen. The molecular weight was determined to be 30. What is the molecular formula? ..... C_2H_6 80

Exercise 8

NAME. . . . . PERIOD . . . . . DATE . . . . .

INSTRUCTOR'S COMMENTS. . . . . .

Chemical Equations

DIRECTIONS: Write the answers to the following in the spaces provided. Make complete statements where appropriate.

1. Why may a formula equation be said to have quantitative significance? . . . . . .

The formula equation indicates the relative mass proportions of reactants and products involved in the reaction.

. 1

2. What important fact concerning a reaction is not revealed by the balanced equation? . . .

The equation reveals nothing of the mechanism by which reactants are converted into products.

. 2

3. What three factors must be considered in writing a balanced chemical equation? . . . . . .

The three factors in equation writing are (1) the equation must represent the facts, (2) it must include the symbols and formulas of all elements and compounds involved as reactants and products, and (3) it must satisfy the law of conservation of atoms.

. 3

4. What are the three steps in an orderly procedure used to write equations? . . . . . .

The three steps in equation writing are (1) represent the facts, (2) write formulas correctly, (3) balance the equation as to atoms.

. 4

5. What are the four general types of chemical reactions? Write the general form equation representing each reaction type.

Type of Reaction	General Form Equations
Composition reaction:	$A + X \rightarrow AX$
Decomposition reaction:	$AX \rightarrow A + X$
Replacement reaction:	$A + BX \rightarrow AX + B$; $Y + BX \rightarrow BY + X$
Ionic reaction:	$A^+ (aq) + B^- (aq) \rightarrow AB$; $AX + BY \rightarrow AY + BX$

. 5

6. What are the six generally recognized types of decomposition reactions? . . . . . .

Metallic carbonates, when heated, form metallic oxides and carbon dioxide.

Many metallic hydroxides, when heated, decompose into metallic oxides and water.

Metallic chlorates, when heated, decompose into metallic chlorides and oxygen.

Some acids, when heated, decompose into nonmetallic oxides and water.

Some oxides, when heated, decompose.

Some decomposition reactions are produced by electricity.

. 6

7. What are the four classes of replacement reactions? .....

Replacement of a metal in a compound by a more active metal.

Replacement of hydrogen in water by metals.

Replacement of hydrogen in acids by metals.

Replacement of halogens. 7

8. What are reversible reactions? .....

Reactions in which the products of a chemical reaction can react and produce the original reactants. 8

9. What are two rules related to the activity series of the elements which help predict the course of replacement reactions? .....

Atoms of a more active metal lose electrons to positively charged ions of a less active metal. Atoms of more active nonmetals acquire electrons from negatively charged ions of less active nonmetals.

.....9

10. What are the four steps used in *setting up* a mass problem for solution by the mole method? .....

(1) Write the balanced equation. (2) Show the problem specifications: what is given and what is required. (3) Show the mole proportions, writing under each substance involved in the problem the number of moles indicated by the equation.

(4) Determine the mass of one mole of each substance involved in the problem.

.....10

11. What are the four main operations which follow the problem set-up in *solving* a mass-mass problem by the mole method? .....

(1) Determine the number of moles of the substance whose mass is given in the problem. (2) Determine the number of moles of the substance whose mass is required. (3) Determine the mass of the substance required. (4) Check the units assigned to make sure they yield the proper units for the answer, and estimate the answer.

.....11

12. How do you convert the given mass of the substance to moles? .....

The given mass is converted to moles by multiplying the mass by the fraction mole/mass of one mole of the substance. 12

13. How do you determine the number of moles of substance whose mass is required?

Multiply the number of moles of the substance whose mass was given by the ratio of the number of moles of substance whose mass is required and the number of moles of substance whose mass is given as indicated by the balanced equation.

.....13

14. How do you determine the mass of the substance required? .....

Multiply the number of moles of the substance whose mass is required by the number of grams per mole of that substance.

.....14

DIRECTIONS: Write the balanced chemical equation in the space provided under each word equation. Identify the type of reaction.

15. sulfur + oxygen → sulfur dioxide (g)

$S + O_2 \rightarrow SO_2$ (g) Composition reaction. 15

16. zinc + sulfuric acid(H_2SO_4) → zinc sulfate (ag) + hydrogen (g)

$Zn + H_2SO_4 \rightarrow ZnSO_4\ (aq) + H_2\ (g)$ Replacement reaction. 16

17. hydrogen + nitrogen → ammonia (g)

$3\ H_2 + N_2 \rightarrow 2\ NH_3\ (g)$ Composition reaction. 17

18. hydrogen + chlorine → hydrogen chloride (g)

$H_2 + Cl_2 \rightarrow 2\ HCl\ (g)$ Composition reaction. 18

19. carbon + water(steam) → carbon monoxide (g) + hydrogen (g)

$C + H_2O \rightarrow CO\ (g) + H_2\ (g)$ Replacement reaction. 19

20. calcium oxide + water → calcium hydroxide (aq)

$CaO + H_2O \rightarrow Ca(OH)_2\ (aq)$ Composition reaction. 20

21. phosphorus + oxygen → diphosphorus pentoxide (molecular formula, P_4O_{10}) (s)

$4\ P + 5\ O_2 \rightarrow P_4O_{10}\ (s)$ Composition reaction. 21

22. hydrochloric acid(HCl) + sodium hydroxide → sodium chloride (aq) + water

$HCl + NaOH \rightarrow NaCl\ (aq) + H_2O$ Ionic reaction. 22

23. barium chloride + sulfuric acid → barium sulfate (s) + hydrochloric acid

$BaCl_2 + H_2SO_4 \rightarrow BaSO_4\ (s) + 2\ HCl$ Ionic reaction. 23

24. aluminum sulfate + calcium hydroxide → aluminum hydroxide (s) + calcium sulfate (s)

$Al_2(SO_4)_3 + 3\ Ca(OH)_2 \rightarrow 2\ Al(OH)_3\ (s) + 3\ CaSO_4(s)$ Ionic reaction. 24

25. copper + chlorine → copper(I) chloride (s)

$2\ Cu + Cl_2 \rightarrow 2\ CuCl\ (s)$ Composition reaction. 25

26. aluminum oxide → aluminum (l) + oxygen (g)

$2Al_2O_3 \rightarrow 4Al(l) + 3O_2(g)$ Decomposition reaction. 26

DIRECTIONS: Complete the word equation and write the balanced formula equation. Identify the type of reaction and give a reason for the product(s). Consult the Activity Series (Section 8.8) and Table 12, Solubilities, in the Appendix as necessary.

27. calcium + oxygen → calcium oxide.

$2Ca + O_2 \rightarrow 2CaO$. Composition, the product is more stable than the reactants. 27

28. copper(II) carbonate → copper(II) oxide + carbon dioxide(g).

$CuCO_3 \rightarrow CuO + CO_2(g)$. Decomposition of a metallic carbonate. 28

29. aluminum + hydrochloric acid → aluminum chloride + hydrogen(g).

$2Al + 6HCl \rightarrow 2AlCl_3 + 3H_2(g)$. Replacement, aluminum is more active than hydrogen. 29

30. potassium iodide + chlorine → potassium chloride + iodine.

$2KI + Cl_2 \rightarrow 2KCl + I_2$. Replacement of halogens. 30

31. barium chloride + sodium sulfate → sodium chloride + barium sulfate(s).

$BaCl_2 + Na_2SO_4 \rightarrow 2NaCl + BaSO_4(s)$. Ionic, barium sulfate precipitates. 31

32. copper + silver nitrate → copper(II) nitrate + silver(s).

$Cu + 2AgNO_3 \rightarrow Cu(NO_3)_2 + 2Ag(s)$. Replacement, copper is more active than silver. .32

33. barium hydroxide + sulfuric acid → water + barium sulfate(s).

$Ba(OH)_2 + H_2SO_4 \rightarrow 2H_2O + BaSO_4(s)$. Ionic, water is formed, barium sulfate precipitates. .33

34. zinc sulfate + ammonium sulfide → ammonium sulfate + zinc sulfide(s).

$ZnSO_4 + (NH_4)_2SO_4 + ZnS(s)$. Ionic, zinc sulfide precipitates. .34

35. sodium + water → sodium hydroxide + hydrogen(g).

$2Na + 2H_2O \rightarrow 2NaOH + H_2(g)$. Replacement, sodium is more active than hydrogen. .35

36. zinc + sulfuric acid → zinc sulfate + hydrogen(g).

$Zn + H_2SO_4 \rightarrow ZnSO_4 + H_2(g)$. Replacement, zinc is more active than hydrogen. .36

37. mercury (II) oxide → mercury + oxygen(g).

$2HgO \rightarrow 2Hg + O_2(g)$. Decomposition, some metallic oxides decompose on heating. .37

DIRECTIONS: Solve the following problems using the method outlined in Chapter 8 of MODERN CHEMISTRY. Place the labeled answers in the spaces provided at the right. Have the solutions of all problems worked out in smooth form on separate paper (or in your problem notebook) to be turned in to your instructor if requested.

38. How many grams of oxygen can be produced by completely decomposing 10 g of mercury(II) oxide? 0.74 g O_2 . . . 38

39. How many grams of mercury are produced in Problem 38? 9.3 g Hg . . . 39

40. An excess of sulfuric acid reacts with 8.10 g of magnesium. (a) How many moles of hydrogen are produced? (a) 0.333 mole H_2 . . . 40a

(b) How many grams of hydrogen are produced? (c) How many grams of magnesium sulfate are produced? (b) 0.671 g H_2 . . . 40b

(c) 40.1 g $MgSO_4$ . . . 40c

41. In a reaction between carbon and oxygen, 11.0 g of carbon dioxide is formed. How many grams of carbon were burned? 3.00 g C . . . 41

42. What mass of copper in grams is precipitated when 13.5 g of aluminum reacts with copper(II) sulfate in solution? 47.6 g Cu . . . 42

43. How many grams of calcium nitrate are produced the reaction between 12.6 g of nitric acid(HNO_3) and an excess of calcium hydroxide? 16.4 g $Ca(NO_3)_2$ . . . 43

44. Suppose that 10.0 g of calcium reacts completely with water. (a) How many moles of hydrogen gas could be collected? (b) How many grams of calcium hydroxide are produced? (a) 0.249 mole H_2 . . . 44a

(b) 18.5 g $Ca(OH)_2$ . . . 44b

Exercise 9

NAME . . . . . PERIOD . . . . . DATE . . . . .

INSTRUCTOR'S COMMENTS . . . . . .

The Gas Laws

DIRECTIONS: In the blank space at the right of each statement, write the word or expression which BEST completes the meaning when substituted for the corresponding number.

1-3. The three basic assumptions of the kinetic theory are that matter is composed of .(1)., the particles of matter are in .(2)., and the particles of matter .(3). in collisions.

very tiny particles 1

continual motion 2

do not lose energy 3

4. The process of spreading out spontaneously to occupy a space uniformly is known as .(4).

diffusion 4

5. Matter in the gaseous phase occupies a volume about .(5). times that which it does in the liquid or solid phase.

1000 5

6-7. If the temperature of a sample of gas within a container is raised, the molecules have more .(6). which is related to the speed of the molecules by the equation .(7).

kinetic energy 6

$E_k = \frac{1}{2} mv^2$ 7

8. The lowest temperature at which a substance can exist as a gas at atmospheric pressure is called the .(8).

condensation temperature. 8

9-12. Substances which consist of low-molecular-weight nonpolar covalent molecules can exist as gases at very .(9). temperatures. Similar substances which have higher molecular weights have condensation temperatures somewhat .(10). room temperature. The condensation temperature of ammonia is much higher than that of methane (a molecule of similar weight and complexity) because the molecule of ammonia is .(11). Ionic compounds, covalent network substances, and metals have condensation temperatures that are all .(12).

low 9

above 10

polar 11

very high 12

13-14. Generally, the higher the molecular weight of a nonpolar molecule, the .(13). its condensation temperature will be. The strength of dispersion interaction forces depends on the number of .(14). in a molecule.

higher 13

electrons 14

15. If the volume which one mole of gas molecules occupies remains constant, the pressure exerted by the gas will .(15). if the temperature is lowered.

decrease 15

16. At a constant temperature, the pressure exerted by one mole of gas molecules will .(16). if the volume available to the gas is increased.

decrease 16

17-18. Standard temperature is exactly .(17). degrees Celsius. Standard pressure is the pressure exerted by a column of mercury exactly .(18). high.

zero 17

760 mm 18

19. Standard pressure is the average atmospheric pressure at .(19).

sea level 19

20-22. The volume of a definite quantity of dry gas is .(20). proportional to the pressure, provided the .(21). remains constant. This statement is known as .(22). law.

inversely 20

temperature 21

Boyle's 22

23-25. The experiments of Jacques Charles revealed that (a) all gases expand or contract at the .(23). rate with changes in temperature, providing the pressure is .(24). and (b) the change in volume amounts to .(25). of the original volume at 0° C for each Celsius degree the temperature is changed.

same 23

constant 24

$\frac{1}{273}$ 25

26. The temperature of the triple point of water is defined as .(26). °K.

273.16 26

27. Kelvin temperatures are just .(27). degrees higher than Celsius temperatures.

273 27

28-29. Charles' law states that the volume of a definite quantity of dry gas varies .(28). with the .(29). temperature, provided the pressure remains constant.

directly 28

Kelvin 29

30. Charles' law holds for real gases with considerable accuracy except at .(30).

low temperatures. 30

DIRECTIONS: Place the properly labeled answers to the following problems in the spaces provided at the right. Use cancellation wherever possible. Observe the rules for significant figures. Show all calculations in your notebook or on a separate sheet of paper. Use the methods outlined in Chapter 9 of MODERN CHEMISTRY.

31. A gas occupies a volume of 75.0 ml when the pressure is 38$\bar{0}$ mm. What volume does the gas occupy at 114$\bar{0}$ mm?

25.0 ml 31

32. The hydrogen in an eudiometer has a volume of 80.0 ml when the pressure is 74$\bar{0}$ mm. What is its volume at standard pressure?

77.9 ml 32

33. If 60$\bar{0}$ ml of hydrogen is collected when the temperature is 27° C, what volume will the gas occupy at 23° C?

592 ml 33

34. The volume of a gas is 1164 ml at 18° C. What is its volume at standard temperature?

1.09×10^3 ml 34

35. To what Celsius temperature must 58$\bar{0}$ ml of oxygen at 17° C be raised to increase its volume to 70$\bar{0}$ ml?

77° C 35

36. Given 70$\bar{0}$ ml of oxygen at 7° C and 80.0 cm pressure, what volume does it have at 27° C and 50.0 cm pressure?

1.20×10^3 ml 36

37. A gas has a volume of 114$\bar{0}$ ml at 37° C and 62$\bar{0}$ mm pressure. Calculate its volume at STP.

819 ml 37

38. A volume of 50.0 ml of gas is collected by displacement of mercury at 17° C and 76$\bar{0}$ mm pressure. The mercury level inside the eudiometer is 30.0 mm higher than that outside. What volume will the gas occupy at STP?

45.2 ml 38

39. A gas occupies a volume of 40.0 ml at 734.8 mm and 22° C. The gas was collected by displacement of water, and the water level inside the eudiometer is the same as that outside. What is the volume of dry gas at STP?

34.8 ml 39

40. A eudiometer contains 45.0 ml of oxygen when the barometer reading is 731.7 mm and the temperature is 27° C. The water level inside the tube is 68 mm higher thant that outside. What volume does the dry oxygen occupy at STP?

37.7 ml 40

Exercise 10

NAME. . . . . PERIOD . . . . . DATE . . . . .

INSTRUCTOR'S COMMENTS . . . . . .

Molecular Composition of Gases

DIRECTIONS: In the blank space at the right of each statement, write the word or expression which BEST completes the meaning when substituted for the corresponding number.

1-2. The law of combining volumes is attributed to .(1). It states that under similar conditions, the volumes of reacting gases and of their gaseous products are expressed in the ratios of .(2).

Gay-Lussac 1

small whole numbers 2

3-4. The law of combining volumes was explained by .(3). This explanation states that under the same conditions of temperature and pressure, equal volumes of all gases contain the same number of .(4).

Avogadro 3

molecules 4

5-11. The reaction between 1 volume of hydrogen and 1 volume of chlorine produces 2 volumes of hydrogen chloride. All molecules of hydrogen chloride have the same composition according to the law of .(5). The molecular proportions for the reaction are .(6). molecule(s) of hydrogen to .(7). molecule(s) of chlorine to .(8). molecule(s) of hydrogen chloride. Therefore, the smallest number of atoms of hydrogen that can be present in each molecule of hydrogen chloride is .(9)., and the smallest number of atoms of chlorine that can be present in each molecule of hydrogen chloride is .(10). The smallest number of atoms per molecule of hydrogen and/or chlorine that satisfy the requirements of the reaction is .(11).

definite composition 5

1 6

1 7

2 8

1 atom 9

1 atom 10

2 atoms 11

12. The word that describes the number of atoms in each molecule of an active gaseous element is .(12).

diatomic 12

13. The word that describes the number of atoms in each molecule of a noble gas is .(13).

monatomic 13

14. At high temperatures, molecules of mercury are .(14).

monatomic 14

15-16. The volume occupied by 1 mole of a gas at STP, is called the .(15). and is .(16). liters.

molar volume 15

22.4 16

17. One mole of O_2 contains .(17). molecules.

6.02×10^{23} 17

18. The gram-molecular weight of a gaseous substance is the mass, in grams, of .(18). liters of the gas measured at STP.

22.4 18

DIRECTIONS: Solve the following problems using the methods outlined in Chapter 10 of MODERN CHEMISTRY. Place the labeled answers in the spaces provided. Have the solutions worked out in smooth form on separate paper or in your notebook.

19. Calculate the mass of 1.00 liter of carbon monoxide, CO, at STP.

1.25 g 19

20. A gaseous compound of uranium has the formula, UF_6. What is the molecular weight of this gas?

352 20

21. Find the density of the gas in Problem 20.

15.7 g/liter 21

22. The density of bromine vapor is 7.15 g/liter. What is the molecular weight of bromine vapor?

160 22

23. At standard conditions, 500 ml of a gas has a mass of 0.995 g. What is the molecular weight of the gas? 44.6 23

24. The mass of 200 ml of acetylene is 0.234 g. What is its molecular weight? 26.2 24

25. A compound contains: carbon, 92.3%; hydrogen, 7.7%. One liter of its vapor has a mass of 3.48 g. What is the empirical formula? CH 25

26. What is the molecular weight of the compound in Problem 25? 78.0 26

27. What is the molecular formula of the compound in Problem 25? C_6H_6 27

28. What is the mass of 1.00 liter of oxygen at STP? 1.43 g 28

29. What is the mass in grams of 250 ml of CO_2 at STP? 0.49 g 29

30. What is the value of the gas constant R calculated from $pV=nRT$? 0.082057 liter. Atom/mole · °K 30

31. Carbon combines with oxygen and forms carbon dioxide gas. How many milliliters of carbon dioxide are produced when 16.5 ml of oxygen is used? 16.5 ml CO_2 31

32. How many milliliters of oxygen are necessary to form 64.5 ml of sulfur dioxide gas (SO_2) by the combustion of sulfur? 64.5 ml O_2 32

33. If 16 liters of carbon monoxide is burned forming carbon dioxide, how many liters of oxygen are required? 8.0 liters O_2 33

34. How many liters of carbon dioxide are produced in Problem 33? 16 liters CO_2 34

35. How many liters of hydrogen chloride are produced when 37 liters of hydrogen is united with chlorine? 74 liters HCl 35

36. If 12 liters of acetylene (C_2H_2) is used, how many liters of oxygen are required for its complete combustion to carbon dioxide and water vapor? 30 liters O_2 36

37. How many liters of carbon dioxide are produced in Problem 36? 24 liters CO_2 37

38. Heptane, C_7H_{16}, is one of the compounds present in gasoline which forms a gas in the automobile engine. This gas unites with oxygen when combustion occurs, forming carbon dioxide and water vapor. How many liters of oxygen are necessary to burn completely 90 liters of hexane vapor? 990 liters O_2 38

39. If air is 21% oxygen, how many liters of air are needed to furnish the necessary oxygen for Problem 38? 4700 liters air 39

40. How many liters of carbon dioxide are formed in Problem 38? 630 liters CO_2 40

41. How many liters of hydrogen are set free when 6.08 grams of magnesium react with hydrochloric acid? 5.60 liters H_2 41

42. How many grams of iron are needed to set free 112 liters of hydrogen from sulfuric acid? Iron(II) sulfate is the other product. 279 g Fe 42

43. How many liters of hydrogen must be used to reduce 119 grams of hot copper(II) oxide? 33.6 liters H_2 43

44. If 1.00 kilogram of water was produced by the reaction of hydrogen and oxygen, how many liters of oxygen were used?

623 liters O_2 44

45. It is determined that 5.6 liters of hydrogen sulfide gas is needed for a particular purpose. How many grams of iron(II) sulfide reacting with hydrochloric acid and forming hydrogen sulfide and iron(II) chloride must be used in the reaction?

22 g FeS 45

46. Sulfur (12.0 g) is burned in oxygen. How many liters of sulfur dioxide gas are produced?

8.37 liters SO_2 46

47. Oxygen (56.0 liters) is combined with phosphorus forming diphosphorus pentoxide. How many grams of phosphorus were used?

62.0 g P_4 47

48. A sample of a vapor having a mass of 0.519 g occupies 123 ml at 10$\bar{0}$ °C and 745 mm. What is its molecular weight?

131 48

49. How many grams of zinc must react with hydrochloric acid in order to produce 250 ml of hydrogen at 2$\bar{0}$° C and 77$\bar{0}$ mm?

0.689 g Zn 49

50. How many liters of oxygen, collected by displacement of water at 22°C and 760 mm, are produced by the decomposition of 5.92 g of potassium chlorate?

1.80 liters O_2 50

Exercise 11

NAME. . . . . PERIOD . . . . . DATE . . . . .

INSTRUCTOR'S COMMENTS . . . . . .

Liquids-Solids-Water

DIRECTIONS: Write the answers to the following in the spaces provided. Where appropriate, make complete statements.

1. What are the five readily observed properties of liquids? (a) definite volume (b) fluidity (c) noncompressibility (d) diffusion (e) evaporation. 1

2. Why is a liquid practically noncompressible? 2

The particles of a liquid are almost as close together as it is possible for them to be.

3. What kind of forces exist between nonpolar molecules?

The relatively weak dispersion interaction forces exist between nonpolar molecules. 3

4. Why are substances, composed of polar molecules, usually liquids at room temperature?

The attractive forces between these molecules are the the combined dispersion interaction and dipole-dipole forces. 4

5. Why is the diffusion of liquids slower than that of gases?

The liquid molecules move more slowly and are closer together, thus their movement is hindered. 5

6. What is meant by a physical equilibrium?

A dynamic state in which two opposing physical changes occur at equal rates in the same system.

6

7. How does raising the temperature of the liquid in a liquid-vapor equilibrium system affect the system? The equilibrium is disturbed and the concentration of vapor molecules above the liquid surface is increased. The increased chance of collision with escaping vapor molecules causes an increase in the rate of condensation. Soon the equilibrium is reestablished at a higher equilibrium vapor pressure. 7

8. On what two factors does the amount of the equilibrium vapor pressure of a liquid depend? It depends on the nature of the liquid and its temperature. 8

9. To what kinds of dynamic equilibria does the principle of Le Chatelier apply?

This principle applies to all kinds of dynamic equilibria. 9

10. What will be the effect of lowering the temperature of an equilibrium system represented by liquid + energy $\rightleftarrows$ vapor?

The reverse reaction is favored and equilibrium will be reestablished at a lower temperature with a reduced vapor concentration. 10

11. The equilibrium system liquid + energy $\rightleftarrows$ vapor has its temperature kept constant but its volume is increased. When equilibrium is restored, how do the following characteristics of the system at the new equilibrium compare in magnitude with the same characteristics in the original equilibrium? (a) vapor pressure the same (b) vapor molecule concentration the same (c) volume of vapor larger (d) number of liquid molecules smaller 11

12. What temperature is the boiling point of a liquid? .....
The temperature at which the equilibrium vapor pressure of the liquid is equal to the prevailing atmospheric pressure. 12

13. During the boiling of a liquid, how do the kinetic energies of the liquid and vapor compare? The kinetic energies of linear motion of the liquid and vapor molecules must be the same. 13

14. What are the two operations involved in liquefying a gas? .....
The gas must be compressed and the heat of compression absorbed. It must then be permitted to expand rapidly. 14

15. Why does compressing a gas always raise its temperature? .....
Energy is acquired by the gas molecules when work is done on them to push them closer together. 15

16. What is meant by the critical temperature of a gas? .....
The critical temperature is the highest temperature at which it is possible to liquefy the gas with any amount of pressure. 16

17. Of what characteristic of molecules if the magnitude of the critical temperature a measure? It is a measure of the attractive forces between molecules. 17

18. What must be done to (a) the temperature of a gas and (b) simultaneously to its pressure in order to liquefy the gas? .....
(a) It must be lowered below the critical temperature. (b) It must be raised to or above the vapor pressure of the liquefied gas at this temperature. 18

19. The critical temperature of sulfur dioxide is 157.2°C while that of water is 374.0°C. What does this indicate about the polarity of the sulfur dioxide molecules compared to that of the water molecules? The sulfur dioxide molecule is less polar than the water molecule. 19

20. What effect does increase in molecular weight have on the critical temperatures of a series of gases whose molecules are nonpolar? .....
The higher the molecular weight of a gas the higher its critical temperature will be. 20

DIRECTIONS: In the blank space at the right of each statement, write the word or expression which BEST completes the meaning when substituted for the corresponding number.

21-23. The observed properties of solids include .(21). shape, .(22). volume, and .(23). diffusion.

definite 21
definite 22
very slow 23

24-27. Solids which have a regular arrangement of particles are .(24). while those having a completely random particle arrangement are .(25).. The particles of a solid are in .(26). motion about their fixed positions. At low temperatures the kinetic energy of the particles is .(27)..

crystalline 24
amorphous 25
weak vibratory 26
small 27

28-31. The freezing of a liquid involves a .(28). of energy by the liquid. The converse physical change, melting, involves a .(29). of .(30). energy by the particles of the solid. The heat energy required to melt one mole of solid at its melting point is its .(31)..

loss 28
gain 29
potential 30
molar heat of fusion 31

32-34. A solid in contact with its vapor can reach an equilibrium, solid + energy ⇄ vapor, and exhibits a characteristic equilibrium .(32)., which, like that of a liquid, depends only on the .(33).. The change of state from a solid to a vapor is known as .(34)..

vapor pressure 32
temperature 33
sublimation 34

35-36. The pattern of points which describes the arrangement of particles in a crystal structure is known as the .(35)., the smallest portion of which determines the pattern of the structure and is called the .(36)..

crystal lattice 35
unit cell 36

37-39. Crystals of common salt, NaCl, belong to the .(37). system. They may form from their solution by the process of .(38).. Crystals that form .(39). will be more nearly perfect.

isometric (cubic) 37
evaporation 38
slowly 39

40-42. As water solidifies it .(40). heat and .(41). one-ninth in volume. Consequently, ice has a density of about .(42). g/cm^3.

releases 40
increases 41
0.9 42

43-44. Liquid water has its maximum density at the temperature .(43).. The boiling temperature of water is raised above 100°C by .(44). the pressure above the boiling water.

4°C 43
increasing 44

45-51. The structural formula for water may be represented as .(45).. The angle between the two hydrogen-oxygen bonds is about .(46). degrees. The H—O bond is a(n) .(47). bond. The bonds in the water molecule do not lie on .(48). and the molecule as a whole is .(49).. Water molecules associate into groups of molecules because of their .(50). and the formation of .(51). bonds.

. 45
105 46
polar covalent 47
the same straight line 48
polar 49
polarity 50
hydrogen 51

52-53. When water is warmed from 0°C two opposing effects occur. Hydrogen bonds .(52)., enabling water molecules to crowd more closely together. The water molecules tend to spread apart as a result of their .(53)..

break 52
increased energy 53

54-62. Water is a very (stable, unstable) .(54). compound. Active metals like sodium react with cold water, setting free hydrogen and forming a(n) .(55). solution. Red hot iron reacts with steam forming hydrogen and .(56).. Ionic oxides of the very active metals react with water and form .(57).. Such oxides are .(58). anhydrides. The oxides of nonmetals such as carbon and sulfur are .(59). compounds, containing .(60). bonds. They unite with water to form a solution containing a(n) .(61).. Such oxides are known as .(62). anhydrides.

stable 54
metallic hydroxide 55
iron oxide 56
soluble hydroxides 57
basic 58
molecular 59
polar covalent 60
acid 61
acid 62

63-65. The name given to such crystals as $CuSO_4 \cdot 5H_2O$ which contain water is .(63).. Since there is always a definite mass of water combined with a definite

hydrates 63

mass of copper(II) sulfate, such crystals illustrate the law of .(64).. The loss of water by such crystals as $Na_2CO_3 \cdot 10H_2O$, on exposure to air, is called .(65)..

definite composition 64

efflorescence 65

66-67. The property of certain substances, such as calcium chloride, to take up water from the air to form a solution, is called .(66).. Common table salt is .(67). because it contains a small amount of magnesium chloride.

deliquescence 66

hygroscopic 67

68-71. The possible combinations of the various isotopes of hydrogen and oxygen result in the existence of .(68). types of water molecules. Water containing atoms of hydrogen-2 has the formula .(69).. It is possible to separate this water from H_2O by .(70).. The most important use of denterium oxide is in .(71).

18 68

D_2O 69

electricity 70

nuclear reactors 71

72-75. Complete the following table for the crystal tapes listed.

Type	Particles composing lattice	Properties	Examples
72. ionic	positive and negative ions	hard, brittle, rather high melting points, good insulators	NaCl, MgF_2
73. covalent network	atoms sharing electrons with neighboring atoms	giant molecules, very hard, brittle, high melting point,	diamond (C_x), $(SiO_2)_x$
74. metallic	lattice of positive ions and electron gas (valence electrons)	hardness and melting point vary over wide range, high electric conductivity	Fe, Cu, Na, Ag
75. covalent molecular	molecules (van der Waals forces and for polar molecules; dipole forces)	low melting points, relatively soft, volatile, good insulators	H_2, O_2, CH_3, CCl_4, C_6H_6, NH_3, H_2O

Exercise 12

NAME . . . . . PERIOD . . . . . DATE . . . . .

INSTRUCTOR'S COMMENTS . . . . . .

The Solution Process

DIRECTIONS: In the blank space at the right of each statement, write the word or expression which BEST completes the meaning when substituted for the corresponding number.

1. A homogeneous mixture of two or more substances, the composition of which may vary within characteristic limits, is known as a(n) .(1).. solution 1

2. Substances which form water solutions that conduct electricity are called .(2).. electrolytes 2

3. Covalent substances that form molecular solutions with water which do not conduct electricity are called .(3).. nonelectrolytes 3

4. Two liquids which are mutually soluble in each other are said to be .(4).. miscible 4

5. A solution in which the dissolved and undissolved solutes are in equilibrium is said to be .(5).. saturated 5

6. The solubility of a solute is defined as the .(6). amount of that solute which can dissolve in a given amount of a certain solvent, under specified conditions. maximum 6

7. The rapid escape of a gas from a liquid in which it is dissolved is called .(7).. effervescence 7

8-9. The attractive forces between gas molecules are .(8). and their motions are relatively .(9).. negligible . . . . . 8 free 9

10. The solubility of a gas in a liquid is limited to its .(10). concentration, under existing conditions. equilibrium 10

11. The solubility of a gas in a liquid is .(11). proportional to the pressure of the gas above the liquid. directly 11

12. The statement of Number 11 is known as .(12). law. Henry's 12

13. Gases which react chemically with their liquid solvents are generally (more, less) .(13). soluble than those gases which do not. more 13

14. In a given mixture of gases, the partial pressure of each gas is proportional to the number of .(14). of that gas. molecules 14

15. The solubility of a gas (increases, decreases) .(15). as the temperature of the solvent is increased. decreases 15

DIRECTIONS: In the space at the right, place the letter or letters indicating each choice which forms a correct statement.

16. The solubility of a solid depends on (a) the pressure on the solvent, (b) the solvent used, (c) the temperature, (d) the nature of the solid. b, c, d 16

17. When a solid is dissolved in a liquid, we would normally expect (a) the temperature of the solution to be lowered, (b) heat to be given up, (c) the temperature of the solution to rise, (d) heat to be absorbed. a, d 17

18. In the case of liquids dissolved in liquids, we would normally expect little change in temperature because (a) only a small amount of solute can dissolve, (b) all liquids are completely miscible, (c) no change in phase occurs, (d) hydration prevents chemical activity.

c 18

19. A solution process which occurs endothermically may take place spontaneously because (a) the attraction of solute particles for solvent particles gives up energy, (b) the entropy change is favorable even though the energy change is unfavorable, (c) both the entropy change and the energy change are favorable, (d) the mixture of solute and solvent represents a more disordered state than that of the unmixed solid and liquid.

b, d 19

20. When the dissolving process is exothermic (a) the heat of solution is said to be negative, (b) the solution usually cools as dissolving proceeds, (c) the heat content of the solution is less than that of its separate components, (d) heating results in increased solubility.

a, c 20

21. The equilibrium system

$KCl(s) + H_2O(l) + \text{heat} \rightleftarrows \text{solution}$

is initially at equilibrium. The addition of heat to the system results in (a) an increase in the rate of the endothermic reaction, (b) faster dissolving than crystallizing until the stress is relieved, (c) an increase in the concentration of KCl in solution, (d) a decrease in the solubility of the solute.

a, b, c 21

22. The equilibrium system solid + $H_2O \rightleftarrows$ heat + solution is initially at equilibrium. The addition of heat to the system results in (a) an increase in the rate of the endothermic reaction, (b) faster dissolving than crystallizing until the stress is relieved, (c) an increase in the concentration of solid in solution, (d) a decrease in the solubility of the solute.

a, d 22

23. The rate at which a solid dissolves in a liquid may be increased by (a) agitating the mixture, (b) using a cooling bath to maintain a constant temperature, (c) powdering the solid, (d) heating the solvent.

a, c, d 23

24. The molality of a solution is an expression (a) of the number of moles of solute per liter of solution, (b) the number of moles of solute per kilogram of solvent, (c) the number of moles of solute per kilogram of solution, (d) the volume of solute per liter of solvent.

b 24

25. The water molecule is frequently referred to as the water dipole because it contains (a) nonpolar covalent bonds unsymmetrically distributed, (b) polar bonds unsymmetrically distributed, (c) polar bonds symmetrically distributed, (d) nonpolar bonds symmetrically distributed.

b 25

26. Hydrogen bonds explain in part the (a) abnormally high boiling point of water, (b) abnormally low freezing point of water, (c) complete miscibility of water and ethyl alcohol, (d) abnormally high vapor pressure of water.

a, c 26

27. The properties of a solvent which are quantitatively decreased by the addition of a solid solute are (a) boiling point, (b) freezing point, (c) vapor pressure, (d) volume.

b, c 27

28. It was found that 85.5 g of a certain nonelectrolyte dissolved in 1 kg of water lowered the freezing point of water 0.465°C. The molecular weight of the solute is (a) 171, (b) 342, (c) 855, (d) 85.5. [b] 28

29-35. The following questions refer to the diagram below which shows vapor pressure of solvent as a function of temperature plotted for a pure solvent and dilute solutions of molalities X and 2X.

29. At any temperature the vapor pressure is highest for (a) pure solvent; (b) solution X; (c) solution 2X. [a] 29

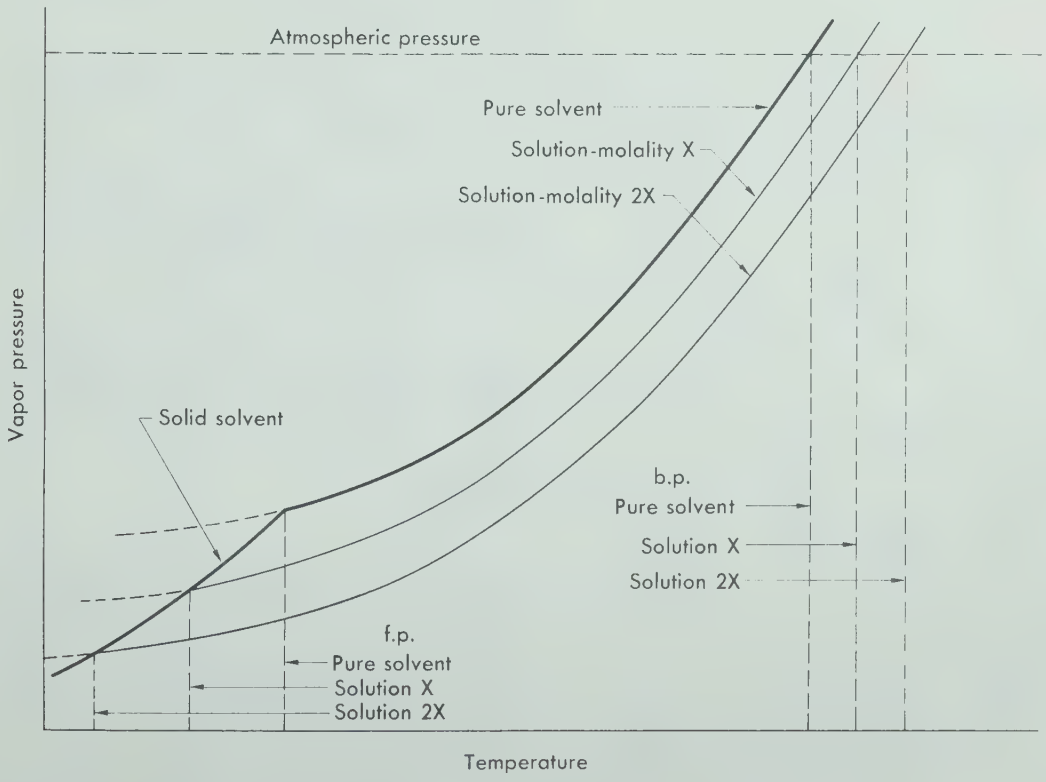
30. The presence of solute particles in solution (a) has no effect on the escaping tendency of solvent molecules; (b) lowers the escaping tendency of the solvent molecules; (c) raises the escaping tendency of the solvent molecules. [b] 30

31. Increasing the concentration of the solution (a) extends the liquid range of the solution; (b) shortens the liquid range of the solution; (c) has no effect on the liquid range of the solution. [a] 31

32. The freezing point is lowest for (a) the pure solvent; (b) solution X; (c) solution 2X. [c] 32

33. When a dilute solution is cooled enough for freezing to occur, the crystals produced are those of the (a) solution; (b) pure solute; (c) pure solvent. [c] 33

34. The boiling point is lowest for (a) the pure solvent; (b) solution X; (c) solution 2X. [a] 34



35. For solutions of equal molality, the effect on the freezing point would be greatest for (a) alcohol; (b) ethylene glycol; (c) common salt. [c] 35

DIRECTIONS: The following questions refer to the solubility curves shown in the graph at left. Write the answers to the questions in the spaces provided.

36. What is the solubility of KNO_3 at 30° C?

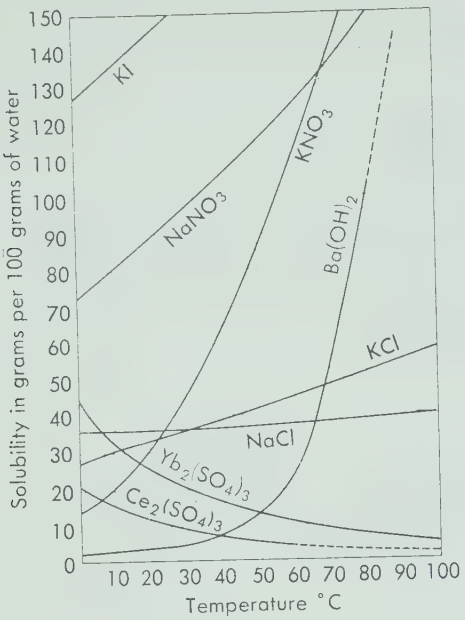
45 g/100 g H_2O 36

37. What is the solubility of KNO_3 at 70° C?

140 g/100 g H_2O 37

38. Which compound varies the least in solubility over the temperature range of the graph?

NaCl 38



39. What change occurs in the solubility of $Ce_2(SO_4)_3$ as the temperature of the solution increases from 20° C to 100° C?

the solubility decreases 39

40. What is the difference between the solubilities of KNO_3 and NaCl at 24°C?

(zero) They are identical 40

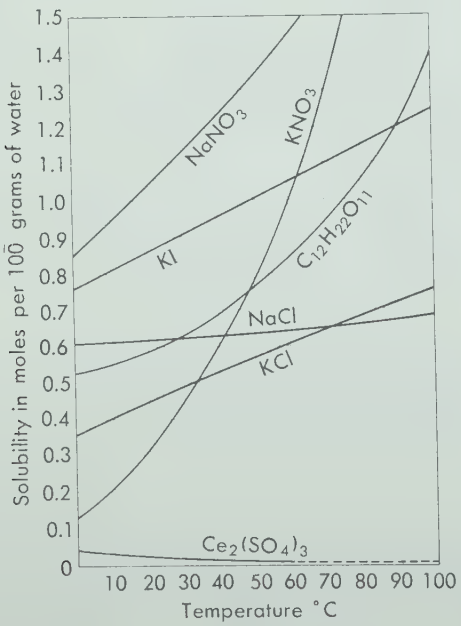
41. What is the difference between the solubilities of KCl and NaCl at 75°C?

12g/100 g H_2O 41

42. What is the average rate of change of solubility of $NaNO_3$ in grams per 100 g of water per Celsius degree in the range from 10° C to 30°C?

0.9 g/100 g H_2O/C° 42

DIRECTIONS: The following questions refer to the solubility curves shown in the graph at the left. Write the answers to the questions in the spaces provided.



43. At what temperature does NaCl have the same solubility in moles per 100 grams of water as sugar ($C_{12}H_{22}O_{11}$)?

28° C 43

44. In terms of solubility in grams per 100 grams of water, how does the solubility of sugar compare with that of NaCl at the temperature which is the correct answer to Question 43?

much greater 44

45. Which pair of compounds has the positions of their *general* solubilities reversed when such positions in the graphs for items 36-42 and 43-45 are compared? (a) KI and $NaNO_3$; (b) $NaNO_3$ and KNO_3; (c) KNO_3 and KCl; (d) KCl and $Ce_2(SO_4)_3$.

a 45

Exercise 13

NAME . . . . . PERIOD . . . . . DATE . . . . . .
INSTRUCTOR'S COMMENTS . . . . . .

Ionization

DIRECTIONS: Write brief answers to the following questions in the spaces provided.

1. What kind of an electric conductor is pure water? — a nonconductor (for all practical purposes) 1

2. What do we call solutes whose water solutions do not conduct electricity? — nonelectrolytes 2

3. What two kinds of substances conduct an electric current when they dissolve in water? — electrovalent substances, polar covalent compounds 3

4. Do electrolytes have a greater or lesser effect on the freezing point of water than nonelectrolytes of equal molal concentration? — greater, 2 to 3 times 4

5. What did Faraday call the particles which carried electricity in water solution? — ions 5

6. How did Arrhenius explain the production of ions? — ionization of molecules of electrolytes in water solution 6

7. How does the total negative ionic charge compare with the total positive ionic charge in the water solution of an electrolyte? — they are equal 7

8. What determines the oxidation number of a simple (one element) ion? — the charge on the simple ion 8

9. How do electrovalent compounds usually exist? — as crystals 9

10. Which end of water dipoles exerts an attractive force on the positive ions of solid sodium chloride? — the negative oxygen end , 10

11. What process occurs when ions separate from the crystals of electrovalent compounds during the solution process? — dissociation 11

12. What name is given to the process by which water molecules attach themselves to the ions of the solute? — hydration 12

13. For the situation represented by the equation, $Na^+Cl^-(s) \rightleftarrows Na^+(aq) + Cl^-(aq)$, how does the tendency toward minimum energy compare with the tendency toward maximum entropy? — they are equal 13

14. What concentrations of aqueous ions are present in water solutions of ionic compounds of very slight solubility? — very low concentrations 14

15. What is the net ionic equation for the reaction which occurs when fairly concentrated solutions of KCl and $AgNO_3$ are mixed? — $Ag^+(aq) + Cl^-(aq) \rightarrow Ag^+Cl^-(s)$ 15

16. Which are the spectator ions in the reaction occurring in Question 15? — $K^+(aq)$ and $NO_3^-(aq)$ 16

17. What effect does melting an electrovalent compound have upon its ions? — ions become mobile and conduct an electric current 17

18. What name is given to the process by which polar solute molecules react with water and form ions? — ionization ... 18

19. Does hydrogen chloride, as a pure liquid, conduct electricity? — no 19

20. What name is given to hydrated protons? — hydronium ions 20

21. Why does a solution of hydrogen chloride in water have decidedly different properties from hydrogen chloride gas? because hydrogen chloride ionizes in water solution . . . 21

22. What is the nature of the bonds in the aluminum halides? covalent with partial ionic character . . . 22

23. How does the conductivity of aluminum chloride in the liquid state compare with its conductivity in water solution? Very poor conductor in liquid state, good conductor in water solution . . . 23

24. What kind of a conductor is a water solution of acetic acid? poor conductor . . . 24

25. To what extent does water ionize? very slightly, about two molecules in a billion . . . 25

26. What type of molecular substances are not acted on by water molecules and do not produce ions? those whose molecules have symmetrical electronic fields . . . 26

27. How is the abnormal lowering of the freezing point of water by hydrogen chloride explained? each molecule of hydrogen chloride which ionizes separates into two particles . . . 27

28. To what extent, compared to the effect of a solution of a nonelectrolyte of the same molality, should a dilute solution of sulfuric acid lower the freezing point of the solvent? 3 times . . . 28

29. What can be found about the ionization of a solute by measuring the lowering of the freezing point by an ionized solute in a measured amount of solvent? the apparent degree of ionization . . . 29

30. The more dilute a sodium chloride solution is, the more nearly the freezing point depression approaches twice the value for a molecular solute. What theory accounts for this fact? the Debye-Hückel theory of attraction between ions . . . 30

DIRECTIONS: In the spaces at the right, indicate which of the following are considered to be electrolytes and which are considered to be nonelectrolytes. Use the symbol E for electrolytes and N for nonelectrolytes.

31. pure water . . . N
32. ethanol (alcohol) . . . N
33. potassium nitrate . . . E
34. glycerol (glycerin) . . . N
35. sodium hydroxide . . . E
36. zinc chloride . . . E
37. nitric acid . . . E
38. copper(II) sulfate . . . E
39. carbon tetrachloride . . . N
40. sucrose (sugar) . . . N

DIRECTIONS: In the spaces provided, write ionic equations for the dissociation of the electrovalent substances in water solution according to the example: $Na^+Cl^-(s) \rightarrow Na^+(aq) + Cl^-(aq)$. Write the equations for ionization of the polar covalent substances in water showing the part played by the water dipoles according to the example:

$$HCl(g) + H_2O(l) \rightarrow H_3O^+(aq) + Cl^-(aq)$$

41. $HBr(g)$ . . . $HBr(g) + H_2O(l) \rightarrow H_3O^+(aq) + Br(aq)$ . . . 41
42. $K_2{}^+SO_4{}^{--}(s)$ . . . $K_2{}^+SO_4{}^{--}(s) \rightarrow 2K^+(aq) + SO_4{}^{--}(aq)$ . . . 42
43. $Zn^{++}(NO_3^-)_2(s)$ . . . $Zn^{++}(NO_3^-)_2(s) \rightarrow Zn^{++}(aq) + 2NO_3^-(aq)$ . . . 43
44. $(NH_4)_3{}^+PO_4{}^{---}(s)$ . . . $(NH_4^+)_3PO_4^{---}(s) \rightarrow 3NH_4^+(aq) + PO_4^{---}(aq)$ . . . 44
45. $K^+OH^-(s)$ . . . $K^+OH^-(s) \rightarrow K^+(aq) + OH^-(aq)$ . . . 45
46. $H_2O(l)$ . . . $H_2O(l) + H_2O(l) \rightleftarrows H_3O^+(aq) + OH^-(aq)$ . . . 46
47. $Al_2Cl_6(s)$ (as for HCl) . . . $Al_2Cl_6(s) + 12H_2O)_6^{+++}(aq) + 6Cl^-(aq)$ . . . 47
48. $H_2SO_4(l)$ (complete ionization) . . . $H_2SO_4(l) + 2H_2O(l) \rightarrow 2H_3O^+(aq) + SO_4{}^{--}(aq)$ . . . 48

Exercise 14

NAME ..... PERIOD ..... DATE .....

INSTRUCTOR'S COMMENTS .....

Acids, Bases, and Salts

DIRECTIONS: In the blank space at the right of each statement, write the word or expression that BEST completes the meaning when substituted for the corresponding number.

1. The organic acid in lemons is .(1). acid.	citric 1
2. Inorganic acids are more commonly known as .(2). acids.	mineral 2
3. The water solutions of the traditional acids are called .(3). acids.	aqueous 3
4. The consumption of .(4). acid is an index to the state of industrialization and prosperity of a country.	sulfuric 4
5. A dye chemist or one who makes explosives would consider .(5). acid to be important.	nitric 5
6. The acid which composes about 0.4% of the human gastric juice is .(6). acid.	hydrochloric 6
7-8. In diluting sulfuric acid the .(7). must be added to the .(8). slowly while stirring.	sulfuric acid ..... 7 water 8
9. The acid whose pure solution is colorless, but which may turn brown on standing is .(9). acid.	nitric 9
10. Concentrated hydrochloric acid is a water solution containing about .(10). % hydrogen chloride.	36 10
11-12. Acids are essentially .(11). bonded structures and have one element in common, .(12).	covalently ..... 11 hydrogen 12
13. Acids ionize in water solution because of the .(13). action of the water dipoles.	hydrating 13
14. The acidic properties of the water solutions of acids are due to the presence of .(14). ions.	hydronium 14
15-16. High concentrations of the ion which is usually represented by the expression .(15). are characteristic of (weak, strong) .(16). acids.	H_3O^+ ..... 15 strong 16
17. When hydrogen chloride is dissolved in a nonpolar solvent, such as toluene, the solution is a (conductor, nonconductor) .(17)..	nonconductor 17
18-19. Hydrogen chloride dissolved in ammonia transfers .(18). to the solvent much as it does in .(19)..	protons ..... 18 water 19
20. According to the Brønsted theory, hydrogen chloride is an acid because it is a(n) .(20). donor.	proton 20
21. Water is a(n) .(21). when gaseous ammonia is dissolved in it.	acid 21
22-23. The strength of an acid depends upon the .(22). in water solution and not upon the amount of .(23). in the molecule.	degree of ionization ..... 22 hydrogen 23
24. The characteristic ion which is present in acid salts of sulfuric acid is the .(24). ion.	hydrogen sulfate, HSO_4^- 24

25-28. Aqueous acids have a .(25). taste, change the color of litmus to .(26)., neutralize .(27)., and react with metals like zinc to release .(28).

29-30. The formula for chloric acid is .(29)., and the formula for chlorous acid is .(30)..

31-33. The oxides formed with nonmetals on the right side of the Periodic Table are, in general .(31). structures which react with water to form .(32). containing one or more .(33).. groups.

34. The formula of the acid anhydride of carbonic acid is .(34).

35. The acid which is used in the production of other acids because it is cheap and has a high boiling point is .(35). acid.

36. Lye is a commercial grade of .(36)..

37-38. Bases are broadly defined as substances which acquire .(37). from another substance. Water solutions of soluble metallic hydroxides are referred to as .(38)..

39. When HCl is dissolved in liquid ammonia, the .(39). acts as the base.

40-41. Hydroxides of active metals furnish .(40). ions in solution, which change the color of phenolphthalein to .(41)..

42-46. When hydroxide ions are added to a solution of an aluminum salt, insoluble .(42). precipitates. An excess of strongly basic OH^- ions causes the precipitate to redissolve and the precipitate acts as a(n) .(43). It also dissolves in an excess of hydronium ions yielding .(44). ions and .(45). Metallic hydroxides which behave in this way are said to be .(46)..

47. The slight ionization of water shows that water is .(47)..

48. The basic ion produced in the slight ionization of liquid ammonia is the .(48). ion.

49. The oxides of active metals are .(49). anhydrides.

50-51. As the oxidation state of the atom combined with the O--H group increases, the bond between this atom and the O--H group becomes more .(50). in character and the removal of an OH^- ion becomes more .(51)..

52. Hydroxides of atoms of high electronegativity and high oxidation states are .(52)..

53. From structural considerations, the molecular formula for sulfuric acid could be written as .(53)..

sour 25

red 26

hydroxides 27

hydrogen 28

$HClO_3$ 29

$HClO_2$ 30

covalent molecular 31

oxyacids 32

hydroxyl 33

CO_2 34

sulfuric 35

sodium hydroxide 36

protons 37

aqueous bases 38

ammonia 39

hydroxide 40

red 41

aluminum hydroxide 42

acid 43

aluminum 44

water 45

amphiprotic 46

amphiprotic 47

amide(NH_2^-) 48

basic 49

covalent 50

difficult 51

acidic 52

$SO_2(OH)_2$ 53

54-55. The species that remains after an acid has given up a proton is called the .(54).. When a base takes on a proton a(n) .(55). is formed.

conjugate base 54

conjugate acid 55

56-58. The HSO_4^- ion is .(56)., since it is *both* the .(57). of SO_4^{--} and the .(58). of H_2SO_4.

amphiprotic 56

conjugate acid 57

conjugate base 58

59-60. The stronger an acid is, the weaker is its .(59).; and the stronger a base, the weaker its .(60)..

conjugate base 59

conjugate acid 60

61-62. According to the Brønsted theory, protolysis reactions favor the production of the (weaker, stronger) .(61). acid and the (weaker, stronger) .(62). base.

weaker 61

weaker 62

63. The solid that remains after the solution resulting from the reaction between zinc and hydrochloric acid is evaporated to dryness, is calssified as a(n) .(63).

salt 63

64. The reaction between CaO and CO_2 produces a compound classified as a(n) .(64)..

salt 64

65-67. A salt is a compound composed of the .(65). of an aqueous base and the .(66). of an aqueous acid. All true salts are .(67). substances.

positive ions 65

negative ions 66

electrovalent 67

68-70. The names of anions(negative ions) of oxyacids take the same root and .(68). as the acid in which they occur. However, the acid ending *-ic* is changed to .(69)., and the ending *-ous* is changed to .(70).

prefix 68

-ate 69

-ite 70

71. Salts from binary acids take the ending .(71)..

-ide 71

DIRECTIONS: Write the required information in the spaces provided.

72. Name the following:

a. NaBr ..... sodium bromide .....

b. $Fe_2(SO_4)_3$ ..... iron(III) sulfate .....

c. $HClO_4$ ..... perchloric acid .....

d. $Ca(HCO_3)_2$ ..... calcium hydrogen carbonate .....

e. $NaClO_3$ ..... sodium chlorate .....

f. Na_2SO_3 ..... sodium sulfite .....

73. State the formulas of the following:

a. chlorous acid ..... $HClO_2$ .....

b. magnesium phosphate ..... $Mg_3(PO_4)_2$ .....

c. ammonium nitrite ..... NH_4NO_2 .....

d. sodium hydrogen carbonate ..... $NaHCO_3$ .....

e. hypochlorous acid ..... HClO .....

f. manganese(IV) chloride ..... $MnCl_4$ .....

74. Write the expression for the conjugate:

a. base of H_2SO_4 ..... HSO_4^- .....

b. base of HCO_3^- ion ..... CO_3^{--} .....

c. base of H_3O^+ ion ..... H_2O .....

d. base of NH_4^+ ion ..... NH_3 .....

e. acid of OH^- ion ..... H_2O .....

f. acid of SO^{--} ion ..... HSO_4^- .....

g. acid of $C_2H_3O_2^-$ ion ..... $HC_2H_3O_2$ .....

h. acid of NH_2^- ion ..... NH_3 .....

75. Write balanced ionic equations for the following reactions:

zinc and an acid (net) $Zn + 2H_3O^+ \rightarrow Zn^{++} + 2H_2O + H_2(g)$

nitric acid and water $HNO_3 + H_2O \rightarrow H_3O^+ + NO_3^-$

hydrogen chloride and ammonia $HCl + NH_3 \rightarrow NH_4^+Cl^-$

ionization of phosphoric acid (first stage) $H_3PO_4 + H_2O \rightarrow H_3O^+ + H_2PO_4^-$

sodium hydroxide and hydrochloric acid (net) $OH^- + H_3O^+ \rightarrow 2\,H_2O$

copper(II) oxide and sulfuric acid (net) $CuO + 2H_3O^+ \rightarrow Cu^{++} + 3H_2O$

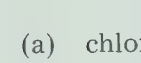
carbon dioxide and water $CO_2 + 2H_2O \rightleftarrows H_3O^+ + HCO_3^-$

ammonia and water $NH_3 + H_2O \rightleftarrows NH_4^+ + OH^-$

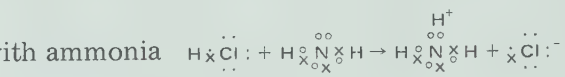
self-ionization of ammonia $NH_3 + NH_3 \rightleftarrows NH_4^+ + NH_2^-$

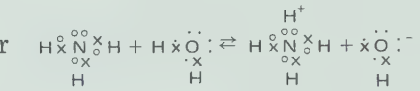
solutions of barium chloride and sodium sulfate $Ba^{++} + 2Cl^- + 2Na^+ + SO_4^{--} \rightarrow 2Na^+ + 2Cl^- + BaSO_4(s)$

76. Write electron-dot formulas for the followngi substances or reactions.

(a) chlorous acid 

(b) sulfuric acid

(c) hydrogen chloride reacting with ammonia 

(d) ammonia reacting with water 

Exercise 15

NAME . . . . . PERIOD . . . . . DATE . . . . .

INSTRUCTOR'S COMMENTS . . . . . .

Acid-Base Titration and pH

DIRECTIONS: In the blank space at the right of each statement, write the word or expression which BEST completes the meaning when substituted for the corresponding number.

1-4. The molality of a solution is an expression of the number of .(1). of solute per .(2). of solvent. For a given solvent, two solutions of equal molality have the same .(3). of solute to solvent .(4)..

5-8. The molarity of a solution is an expression of the number of .(5). solute per .(6). of solution. Equal .(7). of molecular solutions of equal molarity have the same number of .(8)..

9-10. The molecular weight of H_2SO_4 is 98. To make 100 ml of a 1-*M* solution, we would use .(9). g of H_2SO_4 as the solute. To make one liter of its 0.5-*M* solution, we would use .(10) g of H_2SO_4 as the solute.

11. Equivalents are the quantities of substances that have the same .(11). in chemical reactions.

12. KOH has the formula weight 56. In the reaction: $H_2SO_4 + 2KOH \rightarrow K_2SO_4 + 2H_2O$, 49 g of H_2SO_4 is chemically equivalent to .(12). g of KOH.

13-15. For complete neutralizations, one equivalent of a monoprotic acid is the same as .(13). mole of the acid; of a diprotic acid is the same as .(14). mole of acid; of a triprotic acid is the same as .(15). mole of the acid.

16. For the reaction: $H_2SO_4 + NaOH \rightarrow NaHSO_4 + H_2O$, one equivalent of H_2SO_4 is the same as .(16). of the acid.

17. The chemical equivalent of a reactant that is neither an acid nor a base is that quantity in grams, which supplies or acquires 1 mole of .(17). in a chemical reaction.

18. One equivalent of an element can be determined by dividing 1 g-at wt of the element by the change in .(18). taking place during the chemical reaction.

19. The mass of one mole of Na_2SO_4 is 142 g. One equivalent of this salt has a mass of .(19). g.

20-21. The normality of a solution is an expression of the number of .(20). of solute per .(21). of solution.

22-23. One mole of hydrogen chloride has a mass of 36.5 g. One liter of its 0.100-*N* solution contains .(22). g of anhydrous HCl and furnishes .(23). mole of H_3O^+ ions per liter.

moles 1

kilogram 2

ratio 3

molecules 4

moles 5

liter 6

volumes 7

solute molecules 8

9.8 9

49 10

combining capacity 11

56 12

one 13

1/2 14

1/3 15

one mole 16

electrons 17

oxidation state 18

71 19

equivalents 20

liter 21

3.65 22

0.100 23

24-25. A 1-*M* solution of $CuSO_4$ is also a(n) .(24).-*N* A 1-*N* solution of $CuSO_4$ is also a(n) .(25).-*M* solution.

26-32. The self-ionization of water is sometimes referred as .(26). and probably starts with .(27). bond formation. In water at 25°C, the concentration of H_3O^+ ions is .(28). mole of H_3O^+ ions per liter of H_2O. Water is (neutral, acidic, basic) .(29).. The product of the ion concentrations in water is therefore .(30). $mole^2/liter^2$. The principle of .(31). tells us that an increase in $[H_3O^+]$ in an aqueous mixture at equilibrium will cause a(n) .(32). in $[OH^-]$.

33-34. In an acid solution, $[H_3O\ ]$ is (larger, smaller) .(33). than 10^{-7} mole/liter. In an alkaline solution, $[H_3O^+]$ is (larger, smaller) .(34). than 10^{-7} mole/liter.

35-36. Numerically, the pH of a solution is the .(35). of the number of liters of solution that contains .(36). of H_3O^+ ions.

37. The generally useful form of equation for solving for pH is pH = .(37).

38-41. The pH of pure water is .(38).. The pH of a solution which contains 0.00001 mole of H_3O^+ per liter is .(39).. The $[H_3O^+]$ for a solution whose pH is 9, is .(40)., and the solution is (neutral, acidic, alkaline) .(41)..

42-43. Consult Figure 15-3 of the student text. The pH of a solution whose $[H_3O^+]$ is 1.6×10^{-3} is .(42).. The $[H_3O^+]$ of a solution whose pH is 1.3 is .(43)..

44-45. The pH of a 0.01-*M* solution of KOH is .(44).. The pH of a 0.1-*M* solution of $HC_2H_3O_2$ is 2.9. Its $[OH^-]$ is $(1.3 \times 10^{-3}, 7.7 \times 10^{-12})$.(45)..

46-48. The process in which chemically equivalent quantities of H_3O^+ and OH^- ions combine is called .(46).. The process by which the capacity of a solution of unknown concentration to combine with one of known concentration is quantitatively measured is called .(47).. In this process, a dye may serve as an indicator if a color transition takes place within the pH range in which a(n) .(48). point occurs.

49. In the reaction: $HCl + NaOH \rightarrow NaCl + H_2O$, 20.0 ml of the NaOH solution reacts with 10.0 ml of 0.01-*M* HCl solution. The molarity of the NaOH solution is .(49)..

50. In the reaction: $H_2SO_4 + 2NaOH \rightarrow Na_2SO_4 + 2H_2O$, 20.0 ml of the NaOH solution react completely with 10.0 ml of the 0.01-M H_2SO_4 solution. The molarity of the NaOH solution is .(50)..

51. In solving problems to determine the molarity of the aqueous base (or acid), the ratio- moles unknown solute/moles known solute is determined from the .(51).

2 24
0.5 25
autoprotolysis 26
hydrogen 27
10^{-7} 28
neutral 29
10^{-14} 30
Le Chatelier 31
decrease 32
larger 33
smaller 34
common logarithm 35
one mole 36
$-\log[H_3O^+]$ 37
7 38
5 39
1×10^{-9} 40
alkaline 41
2.8 42
0.05-*M* 43
12 44
1.3×10^{-3} 45
neutralization 46
titration 47
equivalence 48
0.005-*M* 49
0.01-M 50
balanced equation 51

52. The equation form which expresses the relationship between volumes and normalities in titration is .(52).. $V_1N_1 = V_2N_2$ 52

53-55. In the reaction: $HC_2H_3O_2 + NaOH \rightarrow NaC_2H_3O_2 + H_2O$, 25.0 ml of 0.100-*N* solution of NaOH reacted with 10.0 ml of the acid. The normality of the acid solution is .(53).. In the reaction: $H_2SO_4 + 2NaOH \rightarrow Na_2SO_4 + 2H_2O$, 25.0 ml of 0.100-*N* solution of NaOH reacted completely with 10.0 ml of the acid. The normality of the acid solution is .(54).. The molarity of this sulfuric acid solution is .(55)..

0.250 *N* 53

0.250 *N* 54

0.125 *M* 55

56-59. In the acid-base titration curves, the pH equivalence point for weak acid-strong base is (lower, higher) .(56). than the equivalence point for strong acid-strong base, a suitable indicator for the weak acid-strong base reaction is (litmus, methyl orange, phenolpthalein) .(57).. The indicator which performs most satisfactorily for a strong acid-weak base titration is (litmus, methyl orange, phenolphthalein) .(58).. For the strong acid-strong base titration, the pH equivalence point has a value of about .(59)..

higher 56

phenolphthalein 57

methyl orange 58

7 59

60. If an indicator added to different solutions assumes the same transition color, the solutions may be considered to have the same .(60)..

pH 60

61. How many milliliters of concentrated HCl are needed to make up 1.00 liter of a 0.200-*N* solution if the concentrated HCl assays 37.4% HCl and has a density of 1.189 g/ml?

16.4 ml 61

62. An approximately 0.02-*N* solution of NaOH when standardized was found to contain 0.0189 mole of OH^- per liter of solution. What was the precise normality of this standard solution?

0.0189 *N* 62

63. How many grams of OH^- ion were contained in each 100 ml of the solution in Question 62?

0.0321 g 63

64. What was the molarity of the solution in Question 63?

0.0189 *M* 64

65. The equivalence point was reached when 30.0 ml of the standard solution of Question 62 was titrated against 20.0 ml of a sulfuric acid solution. What is the normality of the acid solution?

0.0284 *N* 65

66. What is the molarity of the acid solution in question 65?

0.0142 *N* 66

67. Complete the following table.

Substance	Mass of one mole	Mass of 1 equiv
K	39.1 g	39.1 g
Zn	65.4 g	32.7 g
Al	27.0 g	9.0 g
NaBr	102.9 g	102.9 g
$MgCl_2$	95.3 g	47.6 g
H_3PO_4	98.0 g	32.7 g
$Ca(OH)_2$	74.1 g	37.0 g
$Ca_3(PO_4)_2$	310.3 g	51.7 g

Exercise 16

NAME. . . . . PERIOD . . . . . DATE . . . . . .

INSTRUCTOR'S COMMENTS . . . . . .

Carbon and its Oxides

DIRECTIONS: In the blank space at the right of each statement, write the word or expression that BEST completes the meaning when substituted for the corresponding number.

1. The study of carbon compounds is a branch of chemistry called .(1). chemistry.

2-14. Carbon has the atomic number .(2).. Its two .(3). electrons are tightly bound to the nucleus. Its valence electrons are the two .(4). electrons and the two .(5). electrons. Carbon atoms show a very strong tendency to form .(6). bonds. The .(7). valence electrons make it possible to form .(8). covalent bonds. In .(9). hybridization these bonds are directed in space toward the .(10). vertices of a regular .(11).; the .(12). of the atom is at the .(13). of this .(14)..

15. There are several times as many carbon compounds as noncarbon compounds because carbon atoms can .(15)..

16-17. The allotropic forms of carbon are .(16). and .(17)..

18-20. In diamond, each carbon atom is .(18). oriented to its four nearest neighbors. Diamond has a covalent network structure which accounts for its extremely high .(19).. A diamond is a nonconductor of electricity because its electrons are .(20)..

21. Synthetic graphite is made from .(21). in a special furnace.

22-29. The carbon atoms in graphite are arranged in layers of thin .(22). plates. Each carbon atom in a layer is bonded to .(23). other carbon atoms in that layer. The layers of graphite have a(n) .(24). structure in which the carbon-carbon bonds are intermediate in character between .(25). bonds. The layers of carbon atoms are held together by weak .(26). forces accounting for the .(27). of graphite and its .(28). feel. The mobile electrons in a carbon-atom layer make graphite a(n) .(29)..

organic 1

6 2

$1s$ 3

$2s$ 4

$2p$ 5

covalent 6

four 7

four 8

sp_3 9

four 10

tetrahedron 11

nucleus 12

center 13

tetrahedron 14

link together in different ways 15

diamond 16

graphite 17

tetrahedrally 18

melting point 19

not free to migrate 20

petroleum coke 21

hexagonal 22

three 23

resonance 24

single and double 25

dispersion interaction 26

softness 27

greasy 28

conductor of electricity 29

30-31. Graphite is used for the .(30). in electric furnaces electrodes 30
and as a(n) .(31). in electric-arc steel-making furnaces. electrode 31

DIRECTIONS: Write answers to the following in the spaces provided. Where appropriate, make complete statements.

32. What is *destructive distillation?*
The heating of a material in a closed container without access to air or oxygen to decompose it. 32

33. What forms of carbon are produced by destructive distillation?
Destructive distillation produces coke, charcoal, and boneblack. 33

34. What are the volatile byproducts of the destructive distillation of bituminous coal?
The byproducts are coal gas, coal tar, and ammonia. 34

35. Why is coke used in obtaining the metals from the ores of iron, tin, copper, and zinc?
Coke is an excellent reducing agent of high structural strength and freedom from volatile impurities. 35

36. How is activated carbon produced?
Destructive distillation of carbon-containing materials is followed by treatment with steam or carbon dioxide at 100° C. 36

37. How does the porosity of activated carbon account for its ability to adsorb gases?
Porosity accounts for a very large internal surface area. 37

38. What is adsorption? Adsorption is the concentration of a gas, liquid, or solid on the surface of a liquid or solid with which it is in contact. 38

39. What are the best sources for gas-adsorbent activated carbon?
Coconut and other nut shells are the best sources. 39

40. What form of carbon was first used for liquid-phase adsorption?
Boneblack was first used. 40

41. What are the uses of liquid-adsorbent activated carbon? The uses are refining cane sugar, beet sugar, and corn syrup, and removal of objectionable odor and taste in water treatment. 41

42. What type of burning produces soot?
Burning liquid or gaseous fuels composed of carbon and hydrogen in an insufficient supply of air. 42

43. What are the three materials introduced into the furnace in making carbon black?
A spray of liquid fuel or the gaseous fuel vapor, an additional fuel such as natural gas, coke-oven gas, or petroleum refinery gas, air as a source of oxygen. 43

44. Why are the fuels only partially burned in the furnace process for making carbon black?
The fuels are partially burned because the supply of oxygen is insufficient for complete combustion. 44

45. Why is carbon black used in tires? Carbon black preserves the rubber and makes the tire wear longer. 45

46. What is the second largest use of carbon black?
The second largest use of carbon black is in printer's ink. 46

47. How is petroleum coke produced?
Petroleum coke is produced by destructive distillation of oil residues from the refining of crude petroleum. 47

48. To what are rods of petroleum coke converted so that they may be used as electrodes?
The rods are converted to synthetic graphite. 48

49. State two uses of the electrodes described in No. 49. .....

They are used as positive electrodes in dry cells and the production of aluminum by electrolysis. 49

DIRECTIONS: In the blank space at the right of each statement, write the word or expression that BEST completes the meaning when substituted for the corresponding number.

50-52. The amount of carbon dioxide in the atmosphere is about .(50). by volume. Much more carbon dioxide occurs .(51). in the earth's .(52)..

0.03% 50

dissolved 51

surface waters 52

53-57. The common methods of preparing carbon dioxide include: by burning .(53)., by reacting steam and .(54)., by fermentation of .(55)., and by the action of a(n) .(56). on a(n) .(57)..

material containing carbons 53

natural gas 54

molasses 55

acid 56

carbonate 57

58-59. Carbon dioxide is produced by the .(58). of the food we eat. It is also produced by the .(59). of organic matter in nature.

oxidation 58

decay 59

60-63. There are .(60). reactions involved in the usual laboratory method for preparing carbon dioxide. The unstable product of the first reaction is .(61).. Carbon dioxide may be collected either by .(62). or .(63)..

two 60

H_2CO_3 61

water displacement 62

upward air displacement 63

64-65. Carbon dioxide molecules are believed to be resonance hybrids of .(64). electron structures which contribute .(65). to the actual structure.

4 64

equally 65

66-68. Carbon dioxide is a gas at room temperature because of its simple .(66). molecular structure. It is about .(67). dense as air. Because its molecules are heavier than those of oxygen and nitrogen, it diffuses more .(68). than these gases.

nonpolar 66

1.5 times 67

slowly 68

69-70. Solid carbon dioxide is called .(69).. Since its vapor pressure equals atmospheric pressure at $-78.5°C$, solid carbon dioxide .(70). at this temperature.

Dry Ice 69

sublimes 70

71. A piece of burning magnesium continues to burn in a bottle of carbon dioxide because the burning magnesium is hot enough to .(71). the carbon dioxide.

decompose 71

72-73. Carbon dioxide forms a(n) (stable, unstable) .(72). compound with water called .(73)..

unstable 72

carbonic acid 73

74-75. When carbon dioxide is bubbled through limewater, a white precipitate of .(74). is produced. This serves as a test for .(75)..

calcium carbonate 74

carbon dioxide 75

76-77. In the process of .(76)., green plants use carbon dioxide from the air and water from the soil in the presence of sunlight and the catalyst .(77). to produce glucose and oxygen.

photosynthesis 76

chlorophyll 77

78-80. The soda-acid type of fire extinguisher contains separate solutions of .(78). and .(79).. The function of the carbon dioxide produced by their reaction is to .(80)..

sodium hydrogen carbonate, ... 78

sulfuric acid 79

force liquid out of the extinguisher 80

81-82. Carbonated beverages are made by dissolving .(81). under .(82). in flavored water.

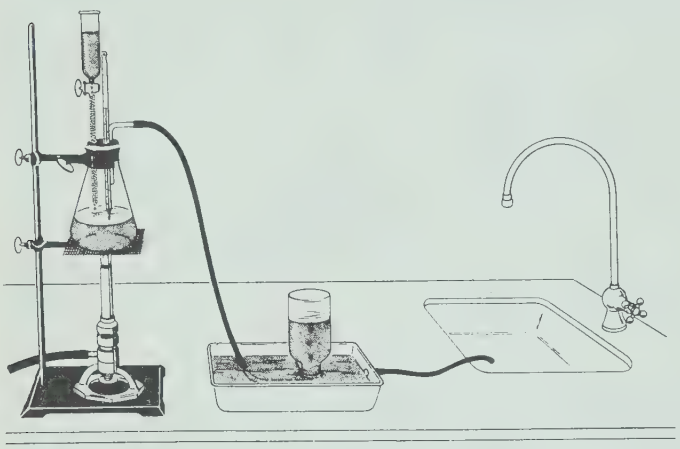
83-85. Yeast produces a(n) .(83). which acts on dough to change some of the .(84). present to ethanol and .(85)..

86-88. Baking powders contain .(86). as a source of carbon dioxide, a substance that produces a(n) .(87). when water is added, and .(88). to keep the powder dry.

89. A useful refrigerant which leaves no liquid residue is .(89)..

carbon dioxide 81
pressure 82
enzymes 83
starches and sugars 84
carbon dioxide 85
sodium hydrogen carbonate 86
acid 87
cornstarch 88
Dry Ice 89

DIRECTIONS: Write answers to the following in the spaces provided. Where appropriate, make complete statements. Questions 90-94 refer to the diagram, which shows a laboratory method of preparing carbon monoxide.



90. What is the liquid in the flask? . Concentrated sulfuric acid . . . 90

91. What is its function in this preparation? . Dehydrating agent 91

92. What is the liquid in the dropping funnel? Formic acid . . . 92

93. Write the equation for the reaction occurring in the flask. . . . . $HCOOH(l) \xrightarrow{H_2SO_4} H_2O(l) + CO_2(g)$ . . . 93

94. What physical property of carbon monoxide is demonstrated by the method of collecting the gas? . Its low solubility in water. . . . 94

95. What properties of carbon monoxide make it particularly hazardous? . . . . . It has no odor, induces drowsiness, is flammable, and is not readily adsorbed by charcoal. . . . 95

96. What use is made of the fact that carbon monoxide reacts readily with oxides at high temperatures? . Carbon monoxide is used to reduce oxide ores. . . . 96

97. What conditions are needed for the reaction between carbon monoxide and hydrogen producing methanol? . Pressure and a mixture of oxides of copper, zinc, and chromium as a catalyst. . . . 97

98-102. Write the equation for the following reactions.

Combustion of CO . . . $2CO(g) + O_2(g) \rightarrow 2CO_2(g)$ . . . 98

Fermentation of sugar . . . $C_6H_{12}O_6(aq) \rightarrow 2C_2H_5OH(aq) + 2CO_2(g)$ . . . 99

Heating $CaCO_3$ . . . $CaCO_3(s) \rightarrow CaO(s) + CO_2(g)$ . . . 100

Carbonate and acid (ionic) . . . $CO_3^{--} + 2H_3O^+ \rightarrow 3H_2O + CO_2(g)$ . . . 101

Calcium hydroxide and CO_2 (ionic) $Ca^{++} + 2OH^- + CO_2 \rightarrow Ca^{++}CO_3^{--}(s) + H_2O$ . . . 102

Exercise 17

NAME. . . . . PERIOD . . . . . DATE . . . . . .
INSTRUCTOR'S COMMENTS . . . . . .

Hydrocarbons

DIRECTIONS: In the parentheses at the right of each word or expression in the second column, write the letter of the expression in the first column which is most closely related.

a. butyl group	C_4H_6	(l)	1
b. carbon-carbon bond in ethane	*p-p*	(f)	2
c. naphthalene	C_4H_9-	(a)	3
d. carbon-carbon internuclear distance in C_2H_2	1.53 Å	(k)	4
e. carbon-hydrogen bond in C_2H_2	sp^2-*s*	(i)	5
f. overlap bond in ethene	sp^3-sp^3	(b)	6
g. carbon-hydrogen bond in ethyne	*sp-sp*	(m)	7
h. anthracene	$C_{10}H_8$	(c)	8
i. carbon-hydrogen bond in ethene	1.33 Å	(j)	9
j. carbon-carbon internuclear distance in ethene	sp^3-*s*	(e)	10
k. carbon-carbon internuclear distance in ethane			
l. butadiene			
m. carbon-carbon bond in ethyne			

---

a. unbreakable bottles and transparent wrappings	sulfur	(g)	11
b. dimerization	benzene	(c)	12
c. coal tar	petroleum	(k)	13
d. styrofoam	naphthalene	(e)	14
e. moth killer	vinyl acetylene	(b)	15
f. coagulation of latex	polystyrene	(d)	16
g. vulcanization	*SBR* rubber	(i)	17
h. neoprene	polyethylene	(a)	18
i. tire treads	acetic acid	(f)	19
j. inner tubes	chloroprene	(h)	20
k. fractional distillation			
l. lead tetraethyl			

DIRECTIONS: In the blank space at the right of each statement, write the word or expression that BEST completes the meaning when substituted for the corresponding number.

21-22. The very large number of carbon compounds which exist is possible because carbon atoms link together with .(21)., and the same number of carbon atoms may be arranged in .(22)..

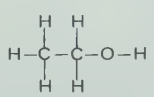
covalent bonds 21

different ways 22

23. Compounds that have the same molecular formula with different structures are called .(23)..

isomers 23

24. Two compounds have the molecular formula C_2H_6O for which only two structural formulas may be written. One compound reacts with sodium liberating one-sixth of its hydrogen. It also reacts with PCl_3 forming the compound C_2H_5Cl. The indicated structure of this compound is .(24)..



. 24

25-26. In a structural formula a dash (—) represents a shared .(25). forming a(n) .(26). bond.

electron pair 25

covalent 26

27-30. Some of the differences between organic and inorganic compounds include: a smaller proportion of organic compounds are soluble in .(27)., organic compounds are usually decomposed by heat .(28). readily, organic reactions proceed at much .(29). rates, and the units of organic compounds are .(30). consisting of atoms joined by covalent bonds.

water 27

more 28

slower 29

molecules 30

31-34. Compounds composed of only the elements hydrogen and carbon are called .(31).. The alkanes have only .(32). bonds. The alkynes have two carbons connected by a(n) .(33). bond, while the alkenes have two carbons connected by a(n) .(34). bond.

hydrocarbons 31

single covalent 32

triple covalent 33

double covalent 34

35-36. The straight- or branched-chain hydrocarbons with two double bonds between carbon atoms are the .(35)., while the aromatic hydrocarbons like benzene have carbon-carbon bonds in which each such bond in the benzene part of the compound is a(n) .(36). bond.

alkadienes 35

resonance hybrid 36

37-38. The members of the alkane series constitute a(n) .(37). series with the general formula .(38)..

homologous 37

C_nH_{2n+2} 38

39. The compound *n*-heptane has a higher melting point and boiling point than *n*-hexane because *n*-heptane has a higher .(39)..

molecular weight 39

40. Isopentane might also be called .(40)..

2-methylbutane 40

41. The alkanes react with halogens such as bromine to form .(41)..

substitution products 41

42. The process of breaking up complex organic molecules into simpler molecules by the action of heat and usually a catalyst is called .(42)..

cracking 42

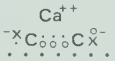
43. The reaction of alkenes with Br_2, H_2, or HBr is called .(43)..

addition 43

44. Molecules of ethene join together at 250 °C and 1000 atmospheres pressure by the process of .(44)..

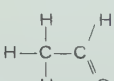
polymerization 44

45. The electron-dot structure for calcium carbide is .(45)..



. 45

46. Ethyne reacts with water from dilute sulfuric acid in the presence of mercury(II) sulfate to yield the compound whose structural formula is .(46)..



. 46

47. Butadiene is prepared by cracking petroleum fractions containing .(47)..

butane 47

48. The benzene ring is sometimes abbreviated and represented by .(48)..

. 48

49. The C_6H_5- group derived from benzene is called the .(49). group.

phenyl 49

50. Bromobenzene is one product formed when benzene reacts with bromine in the presence of iron. This type of reaction is called .(50)..

substitution 50

51. Petroleum refining is a continuous process using a pipe still and a(n) .(51)..

fractionating tower 51

52. Knocking occurs in an automobile engine when the gasoline vapor and air within the cylinders does not burn at a(n) .(52). rate.

uniform 52

53. Gasoline composed of .(53). hydrocarbons tends to knock badly in automobiles.

straight-chain 53

54. The best known gasoline additive used to improve the antiknock properties is .(54)..

lead tetraethyl 54

55. A gasoline which has the characteristics of a mixture of 95% iso-octane and 5% *n*-heptane has an octane rating of .(55)..

95 55

56-57. Thermal cracking of a molecule of $C_{16}H_{34}$ could yield molecules that have eight carbon atoms. Two possible products are .(56). and .(57)..

octane 56

octene 57

58-59. Catalytic cracking produces more molecules of the .(58). and .(59). types.

aromatic 58

branched-chain 59

60. The process used in gasoline production which is the opposite of cracking is .(60)..

polymerization 60

61. The combination of gaseous alkanes with gaseous alkenes in the presence of a sulfuric acid catalyst is .(61)..

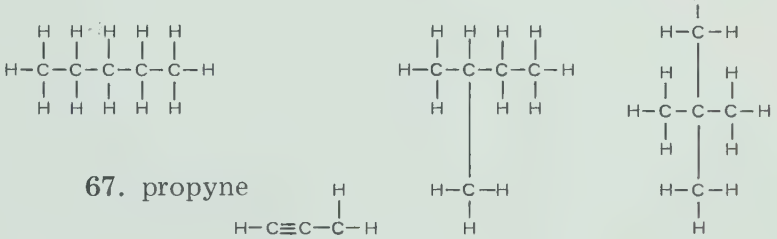
alkylation 61

62. In the process of reforming, .(62). compounds are formed from naphthenes and alkanes.

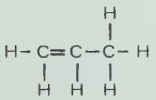
aromatic 62

DIRECTIONS: Draw structural formulas for the following compounds.

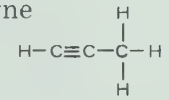
63-65. The three isomers of pentane.



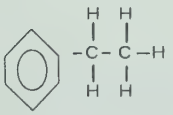
66. propene



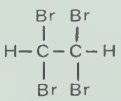
67. propyne



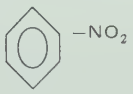
68. ethylbenzene



69. 1,1,2,2, tetrabromoethane



70. nitrobenzene



DIRECTIONS: Write balanced formula equations for the following reactions.

71. Laboratory preparation of methane ..... $NaC_2H_3O_2 + NaOH \rightarrow Na_2CO_3 + CH_4$ 71

72. The action of bromine on methane ..... $CH_4 + Br_2 \rightarrow CH_3Br + HBr$ 72

73. The action of bromine on ethene ..... $C_2H_4 + Br_2 \rightarrow C_2H_4Br_2$ 73

74. The preparation of ethyne from calcium carbide ..... $CaC_2 + 2H_2O \rightarrow Ca(OH)_2 + C_2H_2$ 74

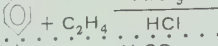
75. The complete combustion of ethane ..... $2C_2H_6 + 7O_2 \rightarrow 4CO_2(g) + 6H_2O$ 75

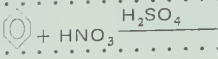
76. A Friedel-Crafts reaction between benzene and RCl (alkyl halogenide) .....

..... $C_6H_6 + RCl \xrightarrow{AlCl_3} C_6H_5R + HCl$ 76

77. The complete combustion of ethyne ..... $2C_2H_2 + 5O_2 \rightarrow 4CO_2 + 2H_2O$ 77

78. Formation of ethene from ethyl alcohol ..... $C_2H_5OH \xrightarrow{H_2SO_4} C_2H_4 + H_2O$ 78

79. Production of ethylbenzene. .....  79

80. Production of nitrobenzene. .....  80

Exercise 18

NAME. . . . . PERIOD . . . . . DATE . . . . . .

INSTRUCTOR'S COMMENTS. . . . . .

Hydrocarbon Substitution Products

DIRECTIONS: In the parentheses at the right of each word or expression in the second column, write the letter of the expression in the first column which is most closely related. The expressions in the first column may be used more than once.

a. RX	acetic acid	(g)	1
b. RMgX	methanol	(c)	2
c. ROH	methyl ethyl ether	(e)	3
d. RCHO	ethyl acetate	(h)	4
e. ROR′	ethyl chloride	(a)	5
f. RCOR′	Grignard reagent	(b)	6
g. RCOOH	acetaldehyde	(d)	7
h. RCOOR′	propionic acid	(g)	8
	ethyl methyl ketone	(f)	9
	carboxyl group	(g)	10
	Fehling's solution	(d)	11
	formyl group	(d)	12

DIRECTIONS: In the blank space at the right of each statement, write the word or expression that BEST completes the meaning when substituted for the corresponding number.

13-15. Alkyl halides may be formed from alkanes by the process of .(13)., from alkenes and alkynes by the process of .(14)., and from an alcohol by treatment with a(n) .(15).. substitution . . . 13; addition . . . 14; hydrogen halide . . . 15

16-18. Alkyl halides react with magnesium and form .(16).. They also react with aqueous solutions of strong hydroxides yielding .(17). and .(18).. Grignard reagents . . . 16; alcohols . . . 17; halide ions . . . 18

19-22. Carbon tetrachloride is prepared commercially by the reaction between .(19). and chlorine. Trichloromethane is commonly called .(20).. CCl_2F_2 is one of a family of compounds called .(21).. Tetrafluoroethene may be polymerized producing the material called .(22).. methane . . . 19; chloroform . . . 20; Freon . . . 21; Teflon . . . 22

23. If ethene is reacted with concentrated sulfuric acid and the mixture is diluted with water, .(23). is produced. ethanol . . . 23

24-25. Fermentation of a dilute solution of sugar or molasses results in the production of the gas, .(24). and of .(25).. CO_2 . . . 24; ethanol . . . 25

26-27. Using a catalyst and high pressure, methanol is synthesized from the gases .(26)., and .(27).. CO . . . 26; H_2 . . . 27

28-29. The molecular formula for ethylene glycol is .(28).. The molecular formula for glycerol is .(29).. $C_2H_4(OH)_2$ . . . 28; $C_3H_5(OH)_3$ . . . 29

30. The reaction of sodium with an alcohol is similar to its reaction with .(30).. water . . . 30

31-32. Ethanol may be dehydrated by hot concentrated sulfuric acid yielding either .(31). or .(32).. diethyl ether . . . 31; ethene . . . 32

33. The oxidation of alcohols with the hydroxyl group

attached to the end carbon in the presence of heated copper(II) oxide produces .(33).. aldehydes 33

34. Sulfation of lauryl alcohol with sulfuric acid and subsequent neutralization with sodium hydroxide yields the very effective detergent whose formula is .(34).. $C_{12}H_{25}OSO_2ONa$ 34

35. Ethers may be synthesized by the action of a sodium alkoxide on a(n) .(35).. alkyl halide 35

36. Acetaldehyde is manufactured commercially by the addition of water to .(36).. ethyne 36

37. The mild oxidation of an aldehyde produces a(n) .(37). having the same number of carbon atoms. organic acid 37

38-39. In the Fehling's test, copper(II) ions are reduced to copper(I) ions, precipitated as .(38). by a(n) .(39).. Cu_2O ..... 38 aldehyde 39

40. The addition of hydrogen to aldehydes, using nickel as a catalyst produces .(40).. alcohols 40

41. Grignard reagents add to aldehydes. Hydrolysis with dilute acid then produces a(n) .(41).. alcohol 41

42. In aldol condensation, one molecule of an aldehyde adds to the .(42). group of the second aldehyde. carbonyl 42

43. Acetone is prepared by the mild oxidation of .(43).. 2-propanol 43

44. The aldol condensation of two molecules of acetone in the presence of barium hydroxide produces the substance called .(44).. diacetone alcohol 44

45. Formic acid may be produced from sodium formate by careful heating with .(45).. sulfuric acid 45

46-50. Carboxylic acids are generally classified as .(46). weak ..... 46 acids. They may be .(47). neutralized ..... 47 by hydroxides just as inorganic acids are. They react with alcohols and form .(48)., esters ..... 48 in whose formation sulfuric acid is used as a(n) .(49). catalytic ..... 49 agent. Experiments with alcohols containing oxygen-18 have shown that the oxygen of the water product comes from the .(50).. acid 50

51-53. The hydrolysis of esters may be carried out in the presence of dilute .(51). or metallic hydroxide solutions. Fats and oils are esters of .(52). and .(53).. acids ..... 51 glycerol ..... 52 long-carbon-chain acids 53

54-56. The hydrolysis of a fat using a solution of a(n) .(54). is called .(55)., producing soaps and .(56).. strong hydroxide ..... 54 saponification ..... 55 glycerol 56

DIRECTIONS: Complete and balance the following equations.

57. $C_{12}H_{22}O_{11} + H_2O \xrightarrow{\text{enzymes}}$ ... $4CO_2 + 4C_2H_5OH$ 57

58. $C_2H_5ONa + CH_3Br \rightarrow$ ... $C_2H_5OCH_3 + NaBr$ 58

59. $CH_3OH + CuO \rightarrow$ ... $HCHO + H_2O + Cu$ 59

60. $C_2H_5OH + O_2 \xrightarrow{\text{enzymes}}$ ... $CH_3COOH + H_2O$ 60

61. Ionization: $CH_3COOH + H_2O \rightleftarrows$ ... $H_3O^+ + CH_3COO^-$ 61

62. $CH_3COOH + C_3H_7OH \rightarrow$ ... $CH_3COOC_3H_2O$ 62

Exercise 19

NAME. . . . . PERIOD . . . . . DATE . . . . .

INSTRUCTOR'S COMMENTS . . . . . .

Reaction Energy and Reaction Kinetics

DIRECTIONS: In the blank space at the right of each statement, write the word or expression that BEST completes the meaning when substituted for the corresponding number.

1-2. Chemical kinetics is concerned with the sequence of steps by which chemical reactions occur, known as the reaction .(1)., and the rates at which they proceed, known as the reaction .(2)..

mechanism 1

velocities 2

3. The energy change in a chemical reaction is directly related to the change in number and strengths of .(3). as the reactants from products.

bonds 3

4-6. In most chemical reactions the energy change can be measured in terms of the .(4). released or absorbed during the reaction. At constant pressure, the heat content is often called the .(5). of the substance and is represented by the symbol .(6)..

heat 4

enthalpy 5

H 6

7. The products of a(n) .(7). reaction have a heat content higher than that of the reactants.

endothermic 7

8. In a thermochemical equation, the .(8). of reactants and products should be shown along with their formulas.

standard states 8

9. Heats of reaction are commonly expressed in terms of .(9). of substance.

kcal/mole 9

10. According to the equation: $H_2O(l) + 68.32$ kcal $\rightarrow H_2(g) + \frac{1}{2} O_2(g)$ when 1 mole of water is decomposed, the products have a heat content which is .(10). by 68.32 kcal.

higher 10

11. The heat of reaction, ΔH, for an exothermic reaction has a(n) .(11). sign.

negative 11

12. Chemical actions in which elements combine and form compounds are generally .(12)..

exothermic 12

13. Compounds with a(n) .(13). heat of formation are very stable.

high negative 13

14. Elements of Groups I and II and Groups VI and VII are far apart in .(14). and in general form stable compounds.

electronegativity 14

15. The heat of combustion, ΔH_c, is defined in terms of 1 mole of .(15)..

reactant 15

16. The heat of formation of compound X equals the sum of heats of formation of products of combustion of compound X minus the heat of .(16). of compound X.

combustion 16

17. The ΔH_f (CO) = −26.41 kcal/mole and the ΔH_c(CO) = −67.64 kcal/mole. The ΔH_f (CO_2) is .(17). kcal/mole.

−94.05 17

18. In the water-gas reaction, the product gases carbon monoxide and hydrogen have collectively a(n) .(18). heat content than the reactants steam and carbon.

higher 18

19. The property which describes the state of disorder of a system is called .(19).. entropy 19

20-21. The property of a system that makes a reaction go consists of two driving forces, a tendency toward the lowest .(20). and a tendency toward the highest .(21).. energy ..... 20 entropy 21

22. A chemical reaction proceeds if it is accompanied by a(n) .(22). in free energy. decrease 22

23-25. An endothermic reaction proceeds only if the action is accompanied by a(n) .(23). in entropy such that $T\Delta S$ is .(24). and .(25). than ΔH. increase ..... 23 positive ..... 24 larger 25

26. In general, a change from a solid to a gas proceeds with a(n) .(26). in entropy. increase 26

27. If all reactants and products are gases, an increase in the number of product particles produces a(n) .(27). in entropy. increase 27

28. Increases in temperature tend to favor .(28). in entropy. increases 28

29-30. When the temperature of a system is low, whether ΔS is positive or negative, the product $T\Delta S$ may be .(29). compared to the ΔH. In such cases the reaction may proceed as the .(30). change predicts. small ..... 29 energy 30

DIRECTIONS: Write the answers to these questions in the spaces provided. Where appropriate, make complete statements.

31. What does a balanced chemical equation reveal about the reaction mechanism? nothing 31

32. What are the four factors or conditions which may affect the rate of a chemical reaction? temperature, concentration of reactants and products, catalysts, nature of reactants, surface area of contact for heterogeneous reactions. 32

33. What technique has been useful in the study of very fast reactions in solution and in gas phase? flash photolysis 33

34. What is meant by a homogeneous chemical system? One in which all reactants and products are in the same phase. 34

35. What are the three equations which represent the complex reaction pathway for the reaction between hydrogen gas and bromine vapor? $Br_2 \rightleftarrows 2Br$; $Br + H_2 \rightleftarrows HBr + H$; $H + Br_2 \rightleftarrows HBr + Br$ 35

36. What are the equations for the two-step mechanism proposed for the reaction $H_2(g) + I_2(g) \rightleftarrows 2HI(g)$? $I_2 \rightleftarrows 2I$; $2I + H_2 \rightleftarrows 2HI$ 36

37. According to the collision theory, in order for reactions to occur between substances (a) what must their particles do, and (b) what must be the result of (a)? (a) Their particles must collide. (b) These collisions must result in interactions. 37

38. According to the collision theory, give two reasons why a collision between reactant molecules may be ineffective. The collision is not energetic enough to supply the required activation energy. The colliding molecules are not oriented in a way that enables them to react with each other. 38

39. What is meant by the "activated complex"? The activated complex is the transitional structure resulting from effective collision and persisting while old bonds are breaking and new bonds are forming. 39

40. How is activation energy related to the activated complex? Activation energy is the energy required to transform the reactants into the activated complex. 40

41. What are the two possibilities to which the activated complex may respond during the brief interval of its existence? The activated complex may re-form the original bonds and separate into reactant molecules, or it may form new bond and separate into product molecules. 41

42. How is the rate of a chemical reaction measured? The rate of reaction is measured by the amount of reactants converted to products in a unit of time. 42

43. According to the collision theory, on what two factors does the rate of a reaction (other than a simple decomposition) depend? The rate of a reaction depends on the collision frequency of the reacting substances and the collision efficiency. 43

44. One factor which influences the rate of chemical reaction is the nature of the reactants. At room temperature, which of the following elements combines most rapidly with oxygen? (a) chlorine; (b) iron; (c) sodium; (d) platinum. (c) sodium. 44

45. In what type of reactions is the reaction rate proportional to the area of contact? The reaction rate is proportional to the area of contact in heterogeneous reactions. 45

46. In the reaction between charcoal and oxygen, the rate of reaction is proportional to (a) the concentration of the solid; (b) twice the concentration of the solid; (c) the concentration of the gas; (d) ten times the concentration of the gas (c) the concentration of the gas. 46

47. How must the specific effect of concentration changes in homogeneous reaction systems be determined? The specific effect of concentration changes must be determined by experiment. 47

48. What possible effects could increasing the concentration of substance A in reacting with substance B have on the reaction rate? It could increase the rate, decrease the rate, or have no effect on the rate. 48

49. When a complex chemical reaction takes place in a series of simple steps, which step determines the rate for the overall reaction? The slowest step which is called the rate-determining step. 49

50. What are the two reasons, according to the collision theory, why the rates of many reactions (at about room temperature) approximately double or triple with a 10 C° rise in temperature? A rise in temperature produces an increase in collision efficiency (since a greater proportion of molecules reach the activation energy) as well as collision frequency. 50

51. How can a catalyst provide an alternate pathway by which the potential energy barrier between reactants and products may be lowered? This could be accomplished by way of an alternate activated complex requiring lower activation energy that somehow involves the catalyst. 51

52. How may the reaction rate for the reaction: $2H_2(g) + 2NO(g) \rightarrow N_2(g) + 2H_2O(g)$ carried out at an elevated constant temperature be measured? The rate may be measured by measuring the decrease in pressure since 4 reactant molecules change to 3 product molecules. 52

53-54. The rate law for the reaction in Question 52 is $R = k[H_2][NO]^2$.

53. What is the specific rate constant for this reaction at a given temperature? The specific rate constant is k. 53

54. What effect does an increase in temperature have on the value of the specific rate constant? Why? A temperature increase usually increases the value of k since an increase in temperature usually increases the rate of reaction. 54

55. What is the simple relationship that exists between the chemical equation for a *step* in a reaction pathway and the rate law for that step? The power to which the molar concentration of each reactant or product is raised in the rate law corresponds to the coefficient for the reactant or product in the balanced equation (for that step). 55

56. If the step involves the reversible reaction $nA + mB \rightleftarrows xC + yD$, what is the rate law for the forward reaction? $R_f = k_f[A]^n[B]^m$ 56

57. Assume that the reaction $2H_2(g) + 2NO(g) \rightarrow N_2(g) + 2H_2O(g)$ occurs in a single step by way of collision between two H_2 molecules and two NO molecules. What would the rate law be, if this assumed reaction mechanism proved to be experimentally correct? $R = k[H_2]^2[NO]^2$ 57

58. The general form for a rate law is $R = k[A]^n[B]^m$ .....If n and m are appropriate powers to which the concentrations must be raised, what determines the value of these powers? Experimental data 58

59. State the law of mass action. The rate of a chemical reaction is directly proportional to the product of the concentrations of reacting substances, each raised to the appropriate power. 59

Exercise 20

NAME . . . . . PERIOD . . . . . DATE . . . . . .

INSTRUCTOR'S COMMENTS . . . . . .

Chemical Equilibrium

DIRECTIONS: In the parenthesis at the right of each word or expression in the second column, write the letter of the expression in the first column which is most closely related.

a.	resistance to change in pH	hydrolysis	(h) 1
b.	opposing processes of equal rate	acid solution	(i) 2
c.	$\frac{[HI]^2}{[H_2]\,[I_2]}$	buffer	(a) 3
d.	$\frac{[HB]\,[OH^-]}{[B^-]}$	ion product constant for water	(e) 4
e.	$[H_3O^+]\,[OH^-]$	equilibrium	(b) 5
f.	$[Ca^{++}]\,[F^-]^2$	an equilibrium constant for a gaseous system	(c) 6
g.	pH greater than 7	alkaline solution	(g) 7
h.	reaction of an ion of a dissolved salt with water	proton donor to water	(j) 8
i.	pH less than 7	hydrolysis constant	(d) 9
j.	$Al(H_2O)_6^{+++}$	a solubility-product constant	(f) 10
k.	pH = 7		
l.	$[C_2H_3O_2^-]\,[H_3O^+]$		

DIRECTIONS: In the space at the right, place the letter or letters indicating each choice which forms a correct statement. More than one response may be correct.

11. When a state of equilibrium is reached between two chemical reactions, (a) all chemical activity ceases, (b) both reactions continue but the net change is zero, (c) the rates of the two reactions are equal, (d) the quantities of reactants and products remain unchanged. b, c, d . . . 11

12. Chemical equilibrium (a) occurs when a solution becomes saturated, (b) is a dynamic state, (c) occurs when a liquid evaporates in an enclosed space, (d) exists when opposing chemical reactions occur at equal rates. b, d . . . 12

13. When an irregularly-shaped crystal of sodium chloride is placed in a saturated solution of sodium chloride (a) the shape of the crystal becomes more irregular, (b) the concentration of the solution increases, (c) the shape of the crystal becomes more regular, (d) the mass of the crystal in contact with the solution decreases. c . . . 13

14. Ionic equilibrium occurs between (a) dissolved molecules and their hydrated ions, (b) undissolved and dissolved solute molecules, (c) a liquid and its vapor in a confined space, (d) undissolved crystals of an electrovalent solute and its dissociated ions. a, d . . . 14

15. In an equilibrium in which the forward reaction runs well toward completion before the speed of the reverse reaction is high enough to establish equilibrium, (a) the products of the reverse reaction are favored, (b) the products of the forward reaction are favored, (c) neither set of products is favored, (d) both reactants and products exist in equal quantities. b . . . 15

16. The rate of a homogeneous reaction may be increased by all of the following except (a) a rise in temperature, (b) the use of a catalyst, (c) an increase in surface area, (d) an increase in concentration. c . . . 16

17. In a reaction between a gas and a solid, the speed of the reaction is proportional to (a) the concentration of the solid, (b) twice the concentration of the solid, (c) the concentration of the gas, (d) twice the concentration of the gas. c . . . 17

18. In a bimolecular reaction between two gases, the speed of the reaction (a) is proportional to the concentrations of both gases, (b) is doubled if the concentrations of both gases are doubled, (c) is doubled if the concentration of one gas is doubled, (d) is independent of the gas concentrations. a, c 18

19. In the equilibrium reaction, $A + B \rightleftarrows C + D$, (a) the speed of the forward reaction increases as A and B are used up, (b) the speed of the reverse reaction increases as A and B are used up, (c) the speed of the forward reaction decreases as A and B are used up, (d) the speeds of both forward and reverse reactions are equal. b, c, d 19

20. In the equilibrium reaction, $A + B \rightleftarrows C + D$, the ratio of the product [C] [D] to the product [A] [B] (a) is always equal to one, (b) is known as the equilibrium constant, (c) may be greater or less than one, (d) depends on the initial concentrations of A and B. b, c 20

21. If the equilibrium constant, K, for a given equilibrium reaction is very much less than one, (a) the original reactants are largely converted to products, (b) the forward reaction is barely underway when equilibrium is established, (c) its numerical value may be changed by increasing the concentration of either reactant, (d) its numerical value may be changed by altering the reaction temperature. b, d 21

22. For the reaction system at equilibrium shown by the equation:

$3A + B \rightleftarrows 2C + 3D$, K is given by the expression

(a) $\dfrac{[2C]\,[3D]}{[3A]\,[B]}$, (b) $\dfrac{[C]\,[D]}{[A]\,[B]}$, (c) $\dfrac{[C]^2\,[D]^3}{[A]^3\,[B]}$, (d) $\dfrac{[C]^3\,[D]^2}{[A]\,[B]^3}$ c 22

23. The expression for the equilibrium constant, K (a) may be written directly from the chemical equation, (b) requires information concerning the kinetics of the system, (c) may be derived from the appropriate rate laws for the forward and reverse reactions, (d) may be calculated from the equilibrium concentrations of reactants and products which are determined experimentally. a, c, d 23

24. For the equilibrium system given by the equation:
$2A_2 + 2BC \rightleftarrows B_2 + 2A_2C$, for which the reaction takes place in two steps
(1) $A_2 + 2BC \rightleftarrows B_2 + A_2C_2$ (slow) (2) $A_2 + A_2C_2 \rightleftarrows 2A_2C_2 \rightleftarrows 2A_2C$ (fast),
(a) $R_f = k_f[A_2][BC]^2$ (for step 1), (b) $R_r = k_r[2C]$ (for step 1),
(c) at equilibrium $R_f = R_r$ (for step 1),
(d) the equilibrium constant for both steps $= \dfrac{[B_2]\,[A_2C]^2}{[A_2]^2\,[BC]^2}$ a, c, d 24

25. For the hypothetical reaction: $A + B \rightleftarrows C + D$, an increase in the concentration of A (a) displaces the equilibrium to the *right*, (b) cause A and B to be used up faster, (c) causes more C and D to be formed, (d) results in a reestablished equilibrium in which the concentration of B will be lower. a, b, c, d 25

26. In the synthesis of ammonia, an increase in pressure results in a greater yield of ammonia because (a) the formation of ammonia molecules tends to relieve the pressure, (b) hydrogen has a lower molecular weight than nitrogen, (c) there are two molecules of ammonia formed for every four molecules of reactants used, (d) ammonia has a lower molecular weight than nitrogen. a, c 26

27. In equilibrium systems in which gases are involved, (a) pressure changes have the effect of altering the concentration of the gases, (b) pressure changes have no effect on the numerical value of the equilibrium constant, (c) an increase in pressure causes a shift in the equilibrium, (d) a decrease in pressure produces an undesirable shift in the equilibrium. a, b 27

28. A change in the operating temperature of an equilibrium reaction (a) always causes a change in the numerical value of the equilibrium constant, (b) favors the endothermic reaction, (c) favors the exothermic reaction, (d) alters the speeds of both reactions unequally. a, d 28

29. The addition of a catalyst to a system (a) does not affect the value of K, (b) accelerates both forward and backward reactions, (c) accelerates the forward reaction more than the backward reaction, (d) reduces the time required for a system to reach equilibrium. a, b, d 29

DIRECTIONS: In the blank space at the right of each statement, write the word or expression which BEST completes the meaning when substituted for the corresponding number.

30. We may drive an equilibrium in a desired direction by the proper application of the principle of .(30).. Le Chatelier 30

31. A reaction between solutions of barium chloride and sulfuric acid may be considered to run to completion because .(31). is practically insoluble. $BaSO_4$ 31

32-33. A reaction between hydrochloric acid and potassium hydroxide may be said to run to completion because .(32). is practically .(33).. water 32

un-ionized 33

34. If a solution of $NaC_2H_3O_2$ is added to a solution of $HC_2H_3O_2$, the concentration of .(34). ions is reduced. H_3O^+ 34

35. The expression for K_a for the ionization of the typical weak acid, HB is .(35).. $\frac{[H_3O^+]\,[B^-]}{[HB]}$ 35

36. A solution of a weak acid may be buffered against changes in .(36). by the addition of a salt of the acid. pH 36

37. The hydronium-ion concentration in water at room temperature is .(37). mole per liter. 10^{-7} 37

38. The hydroxide-ion concentration in water at room temperature is .(38). mole per liter. 10^{-7} 38

39. In any water solution having a pH of 9, the $[OH^-]$ is .(39).. 10^{-5} mole/liter 39

40. If the solution of a salt in water has a pH less than 7, the salt is said to .(40). in water solution. hydrolyze 40

41-43. Aluminum chloride dissolves in water and produces the hydrated cation .(41). which then reacts with water and yields the ions .(42). and .(43).. $Al(H_2O)_6^{+++}$ 41

$Al(H_2O)_5(OH)^{++}$ 42

H_3O^+ 43

44-45. The solubility product constant for the equilibrium system: $BaCO_3(s) \rightleftarrows Ba^{++}(aq) + CO_3^{--}(aq)$ is .(44).. If mixing two solutions results in an ion product which exceeds the K_{sp} of $BaCO_3$, a .(45). will form, $[Ba^{++}]\,[CO_3^{--}]$ 44

precipitate 45

DIRECTIONS: Write the answers to the following problems in the spaces provided. Show calculations in the space below, or in your notebook.

46. The solubility of silver chloride at room temperature is 1.1×10^{-5} mole/liter. What is its K_{sp}? 1.2×10^{-10} 46

47. What is the pH of a 0.001-M solution of NaOH? 11 47

48. Calculate the hydrolysis constanf for an acid whose K_a is 2.0×10^{-5}. 5.0×10^{-10} 48

49. The K_{sp} for FeS is approximately 3.6×10^{-19}. Calculate its solubility in moles/liter. 6.0×10^{-10} 49

50. What is the solubility of FeS in gram/liter of solution? 5.3×10^{-8} 50

Exercise 21

NAME. . . . . PERIOD . . . . . DATE . . . . .

INSTRUCTOR'S COMMENTS . . . . . .

Oxidation-Reduction Reactions

DIRECTIONS: Write the answers to the following in the spaces provided. Make complete statements where appropriate.

1. What is the general definition of oxidation? Oxidation is the loss of electrons by an atom or ion. 1

2. What is the general definition of reduction? Reduction is the gain of electrons by an atom or ion. 2

3. What are the oxidation numbers of the respective elements in NaCl? Explain. . . . . Na = +1, Cl = −1. The oxidation number of a monatomic ion is equal to its charge. NaCl is an ionic compound. 3

4. What are the oxidation numbers of chlorine in Cl_2 and HCl respectively? Explain. . . . . In Cl_2, 0; in HCl −1. In Cl_2, the oxidation number of a free element is zero. In HCl, the Cl is the more electronegative element and the unequally shared electron pair in the polar compound is more strongly attracted to the Cl than to the H. 4

5. What is the oxidation number of each element in (a) nitric acid, HNO_3; (b) copper(II) nitrite, $Cu(NO_2)_2$; (c) ammonium chloride, NH_4Cl; (d) lead(II) chromate, $PbCrO_4$; (e) potassium dichromate, $K_2Cr_2O_7$; (f) nitrogen, N_2; (g) ammonia, NH_3? . . . . . (a) H = +1, N = +5, O = −2; (b) Cu = +2, N = +3, O = −2; (c) N = −3, H = +1, Cl = −1; (d) Pb = +2, Cr = +6, O = −2; (e) K = +1, Cr = +6, O = −1; (f) N = 0; (g) N = −3, H = +1. 5

6. What determines whether a chemical reaction is an oxidation-reduction reaction? . . . . . Reactions in which there is a transfer of electrons either partial or complete are oxidation-reduction reactions. 6

7. What effect does (a) oxidation and (b) reduction have on the oxidation number of a substance? (a) a more positive oxidation state, (b) a more negative oxidation state. 7

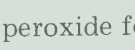
8. What special conservation rule must be observed in balancing oxidation-reduction equations? Conservation of electrons must be observed in addition to the other considerations in balancing oxidation-reduction reactions. 8

9. How do you distinguish between an oxidizing agent and a reducing agent? . . . . . An oxidizing agent is a substance that takes up electrons during a chemical reaction, being reduced in the process. A reducing agent is the substance that furnishes the electrons, being oxidized in the process. 9

10. Why are the Group I elements very active reducing agents? . . . . . The relatively large atoms of Group I have a weak attraction for their valence electrons and form positive ions readily. 10

11. Why are the elements of Group VII very active oxidizing agents? Group VII elements have a strong attraction for electrons and form negative ions readily. 11

12. What is the relationship between the strength of a reducing agent and its corresponding oxidizing agent? The stronger a reducing agent, the weaker is its corresponding oxidizing agent. 12

13. Write the electronic structure which represents the peroxide ion, and state the oxidation number of oxygen in the peroxide form.  = −1. 13

14. What is meant by the chemical equivalent of a reactant? The mass of the substance that acquires or furnishes the Avogadro number of electrons. 14

15. What is the chemical equivalent for tin (atomic weight = 118.7) in the reaction $Sn^{++} - 2e^- \rightarrow Sn^{++++}$? Explain. 59.35 g. Each mole of Sn^{++} ions furnishes two times the Avogadro number of electrons. The gram-equivalent therefore has one-half the mass of a mole. 15

16. Explain the transfer of electrons which takes place in oxidation-reduction reactions. Oxidation-reduction reactions involve the transfer of electrons from the substance being oxidized to the substance being reduced. 16

17. What energy change occurs in spontaneous oxidation-reduction equations when the reactants are in contact? The energy is released during the electron transfer in the form of heat. 17

18. What is an electrochemical cell? An arrangement in which, by separating the reactants in an electrolytic solution, the transfer of electrons may take place through a conducting wire connected between them. 18

19. Which materials serve respectively as (a) cathode and (b) anode in the dry cell? The zinc container is the cathode and the carbon rod is the anode. 19

20. When the external circuit of a dry cell is closed, what reaction occurs at the cathode? $Zn \rightarrow Zn^{++} + 2e^-$ 20

21. Write the equation for the reaction which would occur at the anode of a dry cell (external circuit closed) if MnO_2 were not present. $2NH_4^+ + 2e^- \rightarrow 2NH_3 + H_2(g)$ 21

22. What are electrolytic cells? Electrolytic cells are those in which oxidation-reduction reactions which are not spontaneous may be forced to occur by means of electric energy supplied externally. 22

23. For the electrolysis of water write equations for (a) the net cathode reaction, (b) the net anode reaction, and (c) net reaction for the cell. (a) $2H_2O + 2e^- \rightarrow 2OH^- + H_2(g)$

(b) $6H_2O \rightarrow 4H_3O^+ + O_2(g) + 4e^-$

(c) $2H_2O \rightarrow 2H_2(g) + O_2(g)$ 23

24. For the electrolysis of an aqueous solution of NaCl, write the equations for (a) cathode reaction, (b) anode reaction, and (c) net reaction for the cell. (a) $2H_2O + 2e^- \rightarrow 2OH^- + H_2(g)$

(b) $2Cl^- \rightarrow Cl_2(g) + 2e^-$

(c) $2H_2O + 2Cl^- \rightarrow 2OH^- + H_2(g) +$ 24

$Cl_2(g)$

25. In general, what type of metals yields (a) the metal; (b) hydrogen gas when aqueous solutions of their ions are subjected to electrolysis? . (a) Metals which are difficult to oxidize and form ions which are easily reduced. (b) Metals which are strong reducing agents (easily oxidized) and form ions that are not easily reduced. 25

DIRECTIONS: In the blank space at the right of each statement, write the word or expression that BEST completes the meaning when substituted for the corresponding number.

26-32. A silver plating cell consists essentially of a solution of a(n) .(26)., an anode of .(27)., and a cathode of .(28).. Silver .(29). are .(30). at the cathode while silver .(31). are .(32). at the anode.

soluble silver salt 26
silver 27
the object to be plated 28
ions 29
reduced 30
atoms 31
oxidized 32

33-34. A consistent definition of electrodes defines the electron- .(33). electrode as the cathode, and the electron- .(34). electrode as the anode.

rich 33
poor 34

35-36. The chemical action at the cathode in electrochemical (spontaneous) cells is .(35). while in electrolytic (driven) cells it is .(36)..

oxidation 35
reduction 36

37-42. When the lead storage battery is being charged the cells act as .(37). cells. When it is being discharged, the cells act as .(38). cells. When it is discharging, both electrodes in each cell are converted to .(39). and the electrolyte becomes .(40).. In the electrolyte during charging, .(41). is formed and .(42). is decomposed.

electrolytic 37
electrochemical 38
$PbSO_4$ 39
dilute 40
sulfuric acid 41
water 42

43. In electrochemical cells, we may think of the potential difference as the .(43). of the potentials produced at the cathode and at the anode.

sum 43

44-48. The potential difference between an electrode and its solution in a half-reaction is known as its .(44).. The standard hydrogen electrode consists of a(n) .(45). electrode dipping in a(n) .(46). solution of H_3O^+ ions and surrounded by hydrogen gas at .(47).. It is assigned an electrode potential of .(48). volt(s).

electrode potential 44
platinum 45
1 molal 46
1 atmosphere pressure 47
zero 48

49-50. A copper half-cell coupled with the standard hydrogen electrode gives a potential difference measurement of — 0.34 volt, indicating that copper ions are .(49). readily reduced than hydrogen ions. When a zinc half-cell (standard electron potential = $^-0.76$ volt) is coupled with a copper half-cell, the overall potential difference of the cell is .(50). volt(s).

more 49
+1.10 v 50

DIRECTIONS: Balance each oxidation-reduction equation in the space provided showing the various steps used to arrive at the final equation.

51. copper + nitric acid(dil) → copper(II) nitrate + water + nitrogen monoxide.

STEP 1: $Cu + HNO_3 \rightarrow Cu(NO_3)_2 + H_2O + NO$

STEP 2: $\overset{0}{Cu} + \overset{+\,+5\,-2}{HNO_3} \rightarrow \overset{+2\ \ +5-2}{Cu(NO_3)_2} + \overset{+\ \ -2}{H_2O} + \overset{+2\ -2}{NO}$

STEP 3: $\overset{0}{Cu} - 2e^- \rightarrow \overset{+2}{Cu}$ (oxidation)

$\overset{+5}{N} + 3e^- \rightarrow \overset{+2}{N}$ (reduction)

STEP 4: $3\overset{0}{Cu} - 6e^- \rightarrow 3\overset{+2}{Cu}$

$2\overset{+5}{N} + 6e^- \rightarrow 2\overset{+2}{N}$

STEP 5: $3Cu + 2HNO_3 \rightarrow 3Cu(NO_3)_2 + H_2O + 2NO$

STEP 6: $3Cu + 8HNO_3 \rightarrow 3Cu(NO_3)_2 + 4H_2O + 2NO(g)$

. 51

52. hydrochloric acid + potassium permanganate → manganese(II) chloride + potassium chloride + water + chlorine.

STEP 1: $HCl + KMnO_4 \rightarrow MnCl_2 + KCl + H_2O + Cl_2$

STEP 2: $\overset{+1-1}{HCl} + \overset{+1\ +7\ -2}{KMnO_4} \rightarrow \overset{+2\ \ -1}{MnCl_2} + \overset{+\ \ -1}{KCl} + \overset{+1\ -2}{H_2O} + \overset{0}{Cl_2}$

STEP 3: $\overset{-1}{Cl} - 1e^- \rightarrow \overset{0}{Cl}$ $\quad 2\overset{-1}{Cl} - 2e^- \rightarrow \overset{0}{Cl_2}$ (oxidation)

$\overset{+7}{Mn} + 5e^- \rightarrow \overset{+2}{Mn}$ (reduction)

STEP 4: $10\overset{-1}{Cl} - 10e^- \rightarrow 5\overset{0}{Cl_2}$

$2\overset{+7}{Mn} + 10e^- \rightarrow 2\overset{+2}{Mn}$

STEP 5: $10HCl + 2KMnO_4 \rightarrow 2MnCl_2 + KCl + H_2O + 5Cl_2$

STEP 6: $16HCl + 2KMnO_4 \rightarrow 2MnCl_2 + 2KCl + 8H_2O + 5Cl_2(g)$

. 52

53. aluminum + copper(II) ions → aluminum ions + copper.

STEP 1: $Al + Cu^{++} \rightarrow Al^{+++} + Cu$

STEP 2: $\overset{0}{Al} + \overset{+2}{Cu^{++}} \rightarrow \overset{+3}{Al^{+++}} + \overset{0}{Cu}$

STEP 3: $\overset{0}{Al} - 3e^- \rightarrow \overset{+3}{Al^{+++}}$ (oxidation)

$\overset{+2}{Cu^{++}} + 2e^- \rightarrow \overset{0}{Cu}$ (reduction)

STEP 4: $2\overset{0}{Al} - 6e^- \rightarrow 2\overset{+3}{Al^{+++}}$

$3\overset{+2}{Cu^{++}} + 6e^- \rightarrow 3\overset{+2}{Cu^{++}}$

STEPS 5 & 6: $2Al + 3Cu^{++} \rightarrow 2Al^{+++} + 3Cu$

. 53

54. oxidation of hydroxide ions in electrolytic cell.

STEP 1: $OH^- - e^- \rightarrow H_2O + O_2$

STEP 2: $\overset{-2+1}{OH} \rightarrow \overset{+1\ -2}{H_2O} + \overset{0}{O_2}$

STEP 3: $\overset{-2}{O} - 2e^- \rightarrow \overset{0}{O}$ $\quad 2\overset{-2}{O} - 4e^- \rightarrow \overset{0}{O_2}$ (oxidation)

STEPS 4 & 5: This problem represents oxidation only.

STEP 6: $2OH^- - 4e^- \rightarrow 2H_2O + O_2(g)$

. 54

55. oxidation of lead(II) sulfate during the charging cycle of the lead storage cell.

STEP 1: $PbSO_4 - 2e^- + H_2O \rightarrow PbO_2 + H_3O^+ + SO_4^{--}$

STEP 2: $\overset{+2+6-2}{PbSO_4} + \overset{+1\ -2}{H_2O} \rightarrow \overset{+4\ -2}{PbO_2} + \overset{+1\ -2}{H_3O^+} + \overset{+6-2}{SO_4^{--}}$

STEP 3: $\overset{+2}{Pb} - 2e^- \rightarrow \overset{+4}{Pb}$ (oxidation)

STEPS 4 & 5: This problem represents oxidation only.

STEP 6: $PbSO_4 - 2e^- + 6H_2O \rightarrow PbO_2 + 4H_3O^+(aq) + SO_4^{--}(aq)$

. 55

Exercise 22

NAME. . . . . PERIOD . . . . . DATE . . . . . .

INSTRUCTOR'S COMMENTS . . . . . .

Elements of Period Three

DIRECTIONS: Write answers to the following in the spaces provided. Make complete statements where appropriate.

1. Explain the similarity in properties between elements in a given column in the Periodic Table. They have similar outer-electron arrangements. 1

2. What type of element is the first element in a short period such as Period Three? . . . . . It is a highly reactive metal. 2

3. How do the rest of the elements in Period Three vary in type and character with increasing atomic number? . . . . . In order, there are metals of decreasing activity, a metalloid, nonmetals of increasing activity with the last element being a noble gas. 3

4. Name and state the general characteristics of the element in this period which is classified as a metalloid. . . . . Silicon is the hardest element. It is gray with a metallic luster and has properties between those of metals and nonmetals. 4

5. What are the common physical properties of metals in this period? . . . . . Each is a good conductor of heat and electricity, has a silvery luster, and is ductile and malleable. 5

6. What is the nature of the binding force in metallic crystal lattices? . . . . . It is the attraction between positive ions constituting the lattice and the negative electron gas permeating the lattice. It is essentially uniform in all directions. 6

7. Which of the physical properties of the metals vary with the magnitude of this binding force? . . . . . Variations in the boiling and melting points as well as in degrees of hardness, malleability, and ductility depend on the magnitudes of the forces. 7

8. What does the wide temperature range over which these metals are liquid indicate about the forces existent in the liquid phase? The forces holding the mobile ions together in the liquid phase are evidently quite strong. 8

9. Explain the increase in density as we go from sodium, to magnesium, to aluminum. This increase is explained by the progressively heavier, yet smaller atoms which may be packed closer and closer together. 9

10. In which form does silicon exist in the cubic crystal structure? . . . . . It exists as a covalent network of atoms. 10

11. In terms of electron configuration and covalent bonds, explain that the pattern of arrangement in the silicon crystal is identical with diamond. . . . . The electron configuration of Si is $1s^2\,2s^2\,2p^6\,3s^2\,3p^2$ which is analogous to that of C: $1s^2\,2s^2\,2p^2$. Hybridization occurs in both elements forming four equivalent sp^3 orbitals and hence four equivalent covalent bonds. 11

12. Account for the great difference in melting points between the two consecutive elements aluminum and silicon. The change from metallic to covalent network structure between these two elements accounts for the difference in their melting points. 12

13. Explain the low electric conductivity of silicon.
Some of the valence electrons in silicon crystals are free to move. 13

14. Explain that phosphorus is capable of forming three covalent bonds?
Phosphorus atoms have three half-filled 3*p* orbitals. 14

15. What physical properties of white phosphorus are typical of nonmetals?
It has no luster, is a poor conductor of heat, and a nonconductor of electricity.
15

16. Which characteristics of white phosphorus indicate the existence of only weak dispersion interaction forces between its P_4 molecules?
The low melting and boiling points of white phosphorus together with its brittleness indicate weak dispersion interaction forces. 16

17. Describe the structure of the S_8 molecule.
Each sulfur atom forms two covalent bonds resulting in a molecule of 8 atoms joined together in a puckered ring. 17

18. Explain the relatively low melting point of sulfur.
This is explained by weak dispersion interaction forces between the S_8 molecules. 18

19. Explain that chlorine is a gas at room temperature?
The dispersion interaction forces between the chlorine molecules are very weak. 19

20. Why does argon exist as single atoms?
Argon has no half-filled orbitals to use in bond formation. 20

21. DIRECTIONS: Complete the following table. Use the numbers 1 (smallest) to 8 (largest) to indicate the relative magnitudes of ionization energies and atomic radii and thus the patterns of their variations within the period.

Symbol	Electron configuration	Principal oxidation states	Ionization energy (kcal/ mole atoms)	Atomic radius (Å)
$_{11}$Na	$1s^2 2s^2 2p^6 3s^1$	+1	1	8
$_{12}$Mg	$1s^2 2s^2 2p^6 3s^2$	+2	3	7
$_{13}$Al	$1s^2 2s^2 2p^6 3s^2 3p^1$	+3	2	6
$_{14}$Si	$1s^2 2s^2 2p^6 3s^2 3p^2$	+4	4	5
$_{15}$P	$1s^2 2s^2 2p^6 3s^2 3p^3$	+3, +5	6	4
$_{16}$S	$1s^2 2s^2 2p^6 3s^2 3p^4$	−2, +4, +6	5	3
$_{17}$Cl	$1s^2 2s^2 2p^6 3s^2 3p^5$	−1, +3, +5, +7	7	2
$_{18}$Ar	$1s^2 2s^2 2p^6 3s^2 3p^6$	0	8	1

22. **DIRECTIONS:** Complete the table by filling in the blocks. Identify the type of compound according to the terms: ionic, macromolecular, molecular. Indicate the nature of the reaction with water, if any, in terms of production of OH^- or H_3O^+.

Element	Oxide formula	Type of compound	Nature of reaction with water
Na	Na_2O	ionic	forms OH^-
Mg	MgO	ionic	forms OH^-
Al	Al_2O_3	ionic	none: hydroxide is amphiprotic
Si	SiO_2	covalent network	none
P	P_4O_{10}	molecular	forms $H^+(aq)$
S	SO_2	molecular	forms $H^+(aq)$
Cl	Cl_2O	molecular	forms $H^+(aq)$

DIRECTIONS: In the blank space at the right of each statement, write the word or expression which BEST completes the meaning when substituted for the corresponding number.

23. The outer sublevel electron in aluminum is in a (higher, lower) .(23). energy sublevel than an outer 3*s* electron in magnesium. higher 23

24. A half-filled 3*p* sublevel with three singly occupied orbitals is a (more, less) .(24). stable electron configuration than a 3*p* sublevel with one filled and two singly occupied orbitals. more 24

25. In the presence of hydroxide ion, aluminum hydroxide reacts as a(n) .(25).. acid 25

26. With hot, concentrated solutions containing hydroxide ion, silicon dioxide acts as a(n) .(26).. acid 26

27. Silicon dioxide does not react with common acids but does react with .(27).. hydrofluoric acid 27

28-30. Diphosphorus pentoxide reacts with water forming a solution containing .(28)., whose equilibrium constant for its first ionization is of the order of .(29).. This acid is a(n) .(30). acid.

phosphoric acid 28
10^{-2} 29
moderately strong 30

31-33. Sulfur dioxide reacts with water forming a solution containing .(31)., which is rated as .(32). with an equilibrium constant for its first ionization of the order of .(33)..

sulfurous acid 31
moderately strong 32
10^{-2} 33

34-37. Dichlorine monoxide is prepared by the moderate oxidation of chlorine with .(34).. Its water solution contains .(35)., which is a(n) .(36). acid whose ionization constant is of the order of .(37)..

mercury(II) oxide 34
hypochlorous acid 35
very weak 36
10^{-8} 37

38. Sodium hydride is a(n) .(38). compound. ionic 38

39. The silicon hydrides such as SiH_4 are (more, less) .(39). reactive than the alkanes. more 39

40. Phosphine dissolves in water producing a solution which is .(40).. nearly neutral 40

41. The combustion of hydrogen sulfide produces sulfur or sulfur dioxide and .(41).. water vapor 41

42. The binary hydrogen compound with a period three nonmetal which is completely ionized by water has the formula .(42).. HCl 42

DIRECTIONS: Complete and balance the following equations.

43. $MgO + H_2O \rightarrow$ ..... $Mg^{++} + 2OH^-$ 43

44. $Al(OH)_3 + 3\ H_3O^+ \rightarrow$ ..... $Al(H_2O)_6^{+++}$ 44

45. $SO_2 + 2H_2O \rightleftarrows$ ..... $H_3O^+ + HSO_3^-$ 45

46. $Cl_2O + H_2O \rightarrow$ ..... $2HClO$ 46

47. $NaH + H_2O \rightarrow$ ..... $Na^+ + OH^- + H_2(g)$ 47

48. $SiH_4 + 2H_2O \rightarrow$ ..... $SiO_2 + 4H_2(g)$ 48

49. $H_2S + H_2O \rightleftarrows$ ..... $H_3O^+ + HS^-$ 49

50. $NaCl + H_2SO_4 \rightarrow$ ..... $NaHSO_4 + HCl(g)$ 50

Exercise 23

NAME. . . . . PERIOD . . . . . DATE . . . . . .

INSTRUCTOR'S COMMENTS . . . . . .

The Metals of Group I

DIRECTIONS: In the blank space at the right of each statement, write the word or expression that BEST completes the meaning when substituted for the corresponding number.

1-5. The elements of the Sodium Family are chemically (active, inactive) .(1). and occur in nature only as ions with a(n) .(2). charge. Only trace quantities of the element .(3). have been produced in certain nuclear reactions. The Group I elements form hydroxides which are strongly .(4).. They are relatively soft, have a silvery luster, and are .(5). conductors of electricity and heat.

6-9. The crystal lattice of Group I metals is built around a(n) .(6). unit cell made up of ions with a(n) .(7). charge. The valence electrons form a(n) .(8). that exists throughout the lattice structure. The silvery luster results from the .(9). in the crystal structure.

10. The electron configuration of the sodium ion is .(10)..

11-14. The Group I elements are characterized by .(11). ionization energies. The ionization energy .(12). as the atom size increases going down the group. They are vigorous .(13). agents, the strongest being the lithium atom, probably because of the strong .(14). tendency of its ion.

15-17. These metals are usually stored under .(15). because they react so vigorously with water, releasing .(16). and forming solutions that are strongly .(17)..

18-20. Lithium is prepared by .(18). fused lithium chloride. Crystalline lithium chloride is a vigorous dehydrating agent thereby demonstrating the (high, low) .(19). charge density of the Li^+ ion. The properties of organometallic compounds of lithium suggest that they have .(20). structure.

21-24. Metallic sodium is prepared by the electrolysis of fused .(21). in an apparatus called the .(22).. The other product of this electrolysis is .(23).. Sodium is used in the manufacture of .(24)., the antiknock additive for gasoline.

25-30. The starting material for many other sodium compounds is sodium .(25).. Most commercial sodium hydroxide is produced as a byproduct from the .(26). of a(n) .(27).. Considerable amounts of sodium hydroxide are made by adding calcium hydroxide to .(28). solution. Sodium hydroxide has a destructive effect on skin, hair, and wool and is described as a very .(29). substance. It reacts with .(30). to form soap.

1 . . . active

2 . . . +1

3 . . . francium

4 . . . basic

5 . . . good

6 . . . body centered cubic

7 . . . +1

8 . . . electron gas

9 . . . free electrons

10 . . . $1s^2 2s^2 2p^6$

11 . . . low

12 . . . decreases

13 . . . reducing

14 . . . hydration

15 . . . kerosene (liquid hydrocarbons)

16 . . . hydrogen

17 . . . basic

18 . . . electrolyzing

19 . . . high

20 . . . molecular (covalent)

21 . . . sodium chloride

22 . . . Downs Cell

23 . . . chlorine

24 . . . tetraethyl lead

25 . . . chloride

26 . . . electrolysis

27 . . . aqueous NaCl solution

28 . . . sodium carbonate

29 . . . caustic

30 . . . fats

31-34. In the Solvay process, the substance precipitated is .(31).. It is sold under the common name .(32). or converted into .(33). by thermal decomposition. The substance which is more valuable than these two products and must be recovered for re-use is .(34)..

35-40. The salt produced by the reaction of three moles of NaOH per mole of H_3PO_4 has the formula .(35).. It hydrolzyes in water and the solution is .(36).. Because of this hydrolysis, the .(37). of water is lowered. Many laundry detergents contain the phosphate additive or "builder" whose formula is .(38).. These phosphate builders are essential for good cleaning action because they .(39).. The phosphates in these detergents are of great ecological concern because they contribute to the .(40). of inland lakes and streams.

41-42. The small annual production of potassium is by the reaction between potassium chloride and .(41).. The reaction proceeds to the right because .(42)..

43-46. Potassium hydroxide will neutralize (more, less) .(43). acid than an equal mass of sodium hydroxide. For making black gunpowder, .(44). nitrate is preferred since .(45). is hygroscopic. Sodium compounds (can, cannot) .(46). be substituted for potassium compounds in fertilizers.

47. Of all the metals, electrons are most easily ejected from .(47). by light falling on the metal.

48. When compounds of Group I elements are vaporized, the spectral lines observable through a spectroscope depend on the energy (absorbed, emitted) .(48). by excited electrons returning to their unexcited state.

sodium hydrogen carbonate 31

baking soda 32

sodium carbonate 33

ammonia 34

Na_3PO_4 35

basic 36

surface tension 37

$Na_5P_3O_{10}$ 38

soften water, hold dirt, give an alkaline solution 39

ever-fertilization 40

sodium 41

potassium is removed 42

less 43

potassium 44

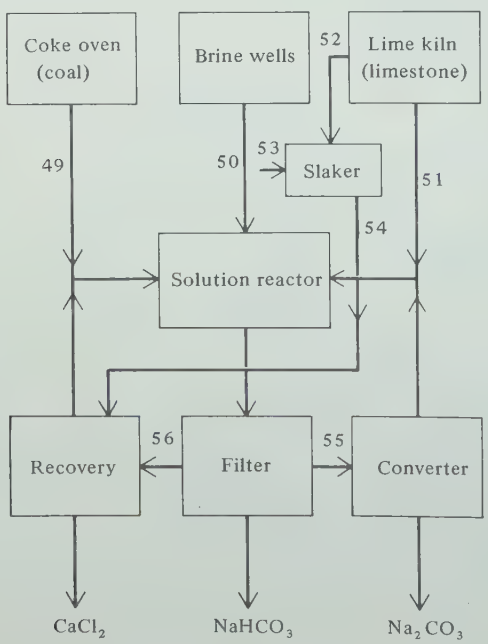
sodium nitrate 45

cannot 46

cesium 47

emitted 48

DIRECTIONS: In the flow chart for the Solvay Process given below, the formulas for substances involved have been omitted. In the corresponding numbered spaces at the right, supply the proper formulas.



NH_3 49

NaCl 50

CO_2 51

CaO 52

H_2O 53

$Ca(OH)_2$ 54

$NaHCO_3$ 55

NH_4Cl 56

DIRECTIONS: Write the balanced chemical equations for:

57. Solvay Process: the net reaction taking place in the production and precipitation of $NaHCO_3$ . . . . . $CO_2(g) + NH_3(g) + H_2O + Na^+(aq) \rightarrow NH_4^+ + NaHCO_3(s)$ 57

58. Solvay Process: the reaction producing Na_2CO_3 . . . . . $2NaHCO_3(s) \rightarrow Na_2CO_3(s) + H_2O(g) + CO_2(g)$ 58

59. Solvay Process: release of ammonia from the ammonium ion . . . . . $NH_4^+(aq) + OH^-(aq) \rightarrow NH_3(g) + H_2O$ 59

60. Hydrolysis of the carbonate ion . . . . . $CO_3^{--} + H_2O \rightleftarrows HCO_3^- + OH^-$ 60

61. The reaction between sodium and water . . . . . $2Na(s) + 2H^2O \rightarrow 2Na^+(aq) + 2OH^-(aq) + H_2(g)$ 61

62. The reaction of slaked lime with sodium carbonate solution . . . . . $Ca(OH)_2 + Na_2CO_3 \rightarrow 2NaOH + CaCO_2(s)$ 62

63. The reaction of calcium with fused rubidium chloride $Ca + 2RbCl \rightarrow CaCl_2 + 2Rb(g)$ 63

64. The cell reaction for the electrolysis of an aqueous NaCl solution . . . . . $2H_2O + 2Cl^- \rightarrow H_2(g) + Cl_2(g) + 2OH^-(aq)$ 64

65. Hydrolysis of PO_4^{---} ion. . . . . $PO_4^{---} + H_2O \rightarrow HPO_4^{--} + OH^-$ 65

Exercise 24

NAME. . . . . PERIOD . . . . . DATE . . . . .

INSTRUCTOR'S COMMENTS . . . . . .

The Metals of Group II

DIRECTIONS: Write the answers to the following questions in the spaces provided. Make complete statements where appropriate.

1. Why are the members of the Calcium Family more dense, harder, and with higher melting and boiling points than the members of the Sodium Family? There is a stronger force of attraction between the doubly-charged metal ions and the electron cloud of the metallic crystals.

2. Why are calcium atoms smaller than potassium atoms? Calcium ions have a higher nuclear charge resulting in smaller K, L, and M shells.

3. Why does strontium form peroxides with oxygen? Because of the large size of its ions.

4. Why are hydroxide solutions of calcium and magnesium weakly basic? Although ionic, they are only slightly soluble in water.

5. How is beryllium isolated? By electrolysis of a fused mixture of sodium and beryllium chlorides.

6. What are two uses of beryllium-copper alloys? They are used for nonsparking tools and practically unbreakable springs.

7. What two properties of beryllium compounds indicate the appearance of nonmetallic properties in this Group? They are somewhat covalent and are extensively hydrolyzed in water.

8. What is the most important source of magnesium? Sea water.

9. How is magnesium produced from (a) magnesium chloride and (b) magnesium oxide?

(a) Electrolysis of fused $MgCl_2$

(b) Reduction by ferrosilicon.

10. Why is magnesium said to be a self-protective metal? It forms a nonporous, nonscaling coat of tarnish (basic magnesium carbonate) in moist air.

11. What are the two products of the combustion of magnesium in air? MgO and Mg_3N_2.

12. With what metal does magnesium form light, strong alloys? Aluminum.

13. How is chemical reduction employed today for recovering calcium? Calcium oxide is heated with aluminum in a vacuum retort. The calcium metal is distilled off as a gas at the high reduction temperature.

14. In what *natural* forms does calcium carbonate exist? Limestone, calcite, marble, shells,

15. What are the two principal raw materials for making cement? Limestone and clay.

16. What metallic ions commonly cause water to be hard? Ca^{++}, Mg^{++}, (Fe^{++}).

17. Why may water that contains the metallic ions in Question 17 with (a) HCO_3^- ions be softened by boiling wheras with (b) SO_4^{--} ions it cannot? (a) These hydrogen carbonates decompose and form insoluble carbonates. (b) The sulfates are not decomposed by heating. 17

18. How does the addition of a basic solution to temporary hard water soften it? The OH^- ions neutralize the HCO_3^- ions and precipitate the metal ions as carbonates. 18

19. How is a zeolite water softener regenerated? By immersing the exhausted zeolite containing Ca zeolite in a sodium chloride solution, the Ca^{++} ions are replaced. 19

20. What principle is employed in the regeneration of a zeolite water softener? The law of mass action. 20

21. How may a synthetic base-exchange resin be regenerated? By using a basic solution. 21

22. Why is it necessary to sweep out the CO_2 from the kiln in which CaO is produced? To avoid the concentration of CO_2 which would drive the reaction in the reverse direction according to Le Chatelier's principle. 22

23. How does lime mortar harden? The first step is the loss of water by evaporation. The lime and sand react slowly forming $CaSiO_3$ and H_2O. CO_2 from the air also reacts with the lime forming $CaCO_3$. 23

24. How is plaster of Paris produced? By gentle heating of gypsum resulting in partial dehydration. 24

25. What compound is used in making red light for fireworks? Strontium nitrate. 25

26. What is one use for barium nitrate? For producing yellow-green color in fireworks. 26

DIRECTIONS: Write equations for these reactions:

27. Burning magnesium in carbon dioxide $2Mg + CO_2 \rightarrow 2MgO + C$ 27

28. Reaction of calcium with water $Ca + 2H_2O \rightarrow (Ca(OH)_2 + H_2(g)$ 28

29. Production of precipitated chalk $Na_2CO_3 + CaCl_2 \rightarrow CaCO_3(s) + 2\ NaCl$ 29

30. Electrolysis of fused magnesium chloride $Mg^{++} + 2Cl^- \rightarrow Mg + Cl_2(g)$ 30

31. Reaction of soap with calcium ions $Ca^{++} + 2C_{18}H_{35}O_2^- \rightarrow Ca(C_{18}H_{35}O_2)_2(s)$ 31

32. Boiling temporary hard water $Ca^{++} + 2HCO_3^- \rightarrow CaCO_3(s) + H_2O + CO_2(g)$ 32

33. Sodium zeolite and calcium ions $Ca^{++} + Na_2$ zeolite $\rightarrow$ Ca zeolite $+ 2Na^+$ 33

34. Calcium oxide and water $CaO(s) + H_2O \rightarrow Ca(OH)_2(s)$ 34

35. Heating calcium carbonate in a kiln $CaCO_3(s) \rightarrow CaO(s) + CO_2(g)$ 35

36. Slaked lime and carbon dioxide $Ca(OH)_2 + CO_2 \rightarrow CaCO_3 + H_2O$ 36

Exercise 25

NAME . . . . . PERIOD . . . . . DATE . . . . . .

INSTRUCTOR'S COMMENTS . . . . . .

The Transition Metals

DIRECTIONS: In the blank space at the right of each statement, write the word or expression which BEST completes the meaning when substituted for the corresponding number.

1. In the fourth period beyond calcium, the elements whose inner 3*d* sublevel is occupied by electrons before the occupation of the fourth energy level is resumed are called the .(1). elements. transition 1

2. In the sixth period the expansion of the 4*f* orbitals gives rise to the .(2). Series. Lanthanide 2

3-7. The important properties of the transition elements are their distinctly .(3). character, .(4). oxidation state(s), strong .(5)., tendency to form .(6). ions, and attraction into a(n) .(7). field. metallic . . . . . 3; variable . . . . . 4; color . . . . . 5; complex . . . . . 6; magnetic 7

8. Within the 6th, 7th, and 8th subgroups, the similarities across each period are .(8). than those down each subgroup. greater 8

9-10. The Fe^{++} ion is formed by the removal of .(9). electron(s) from the Fe atom. The Fe^{+++} ion is formed from the Fe^{++} ion by the further removal of a(n) .(10). electron. two 4*s* 9; 3*d* 10

11-13. The ferromagnetic nature of Fe, Co, and Ni is thought to be related to the similarities in the .(11). orientation of their .(12). 3*d* electrons. In these metals, groups of atoms may be so aligned as to form a small magnetized region, called a(n) .(13).. spin . . . . . 11; unpaired . . . . . 12; domain 13

14-15. The great deposits of the rich iron ore, .(14)., have been depleted. The iron industry now depends on lower grade ores, the most abundant of which is .(15).. hematite . . . . . 14; taconite 15

16-19. The reducing agent that actually reduces the iron ore in the blast furnace is .(16).. The silica impurity combines with the .(17). produced from the added .(18). to form the slag whose formula is .(19).. carbon monoxide . . . . . 16; calcium oxide . . . . . 17; limestone . . . . . 18; $CaSiO_3$ 19

20-21. In the basic-oxygen process for producing steel, the element .(20). is blown in. It combines with .(21). and other unwanted elements. oxygen . . . . . 20; carbon 21

22-23. In the electrochemical explanation of corrosion of iron, the positive electrode in miniature electrochemical cells consists of .(22). and the negative electrode consists of .(23).. carbon . . . . . 22; iron 23

24-25. The ion identified by the formation of a deep-blue precipitate in a solution to which $K_3Fe(CN)_6$ has been added is the .(24). ion. The composition of this precipitate is considered to be .(25).. Fe^{++} . . . . . 24; $KFeFe(CN)_6 \cdot H_2O$ 25

26-29. In the electrolytic refining of copper, the two metals .(26). are not oxidized at the .(27).. The only ions reduced at the .(28). are the .(29). ions.

silver and gold 26
anode 27
cathode 28
Cu^{++} 29

30-31. The addition of an excess of ammonia to a solution of copper(II) ions results in a .(30). color due to the formation of the .(31). ion.

deep-blue 30
$Cu(NH_3)_4^{++}$ 31

32. Copper reacts vigorously with cold .(32). acid.

nitric 32

33. In the cyanide process, silver is precipitated from the filtered sodium argenticyanide solution by the addition of .(33)..

zinc 33

34-36. The addition of hot water to a mixed precipitate of the chlorides of silver, lead, and mercury(I) dissolves out the .(34). chloride. Washing the residue with ammonia-water converts the .(35). chloride to the soluble complex .(36). ion.

lead 34
silver 35
$Ag(NH_3)_2^+$ 36

37-39. The gold from low-grade ores is recovered by the .(37). process. Soluble $AuCl_3$ may be produced by treating gold with moist .(38). or with .(39)..

cyanide 37
chlorine gas 38
$AuCl_4^-$ ions 39

40-42. The mercury(I) ion exhibits an oxidation number of .(40)., yet is known to have the structure .(41). resulting from the formation of a(n) .(42). bond between two Hg^+ ions.

+1 40
Hg_2^{++} 41
covalent bond 42

43-46. The zinc ore, sphalerite, is first roasted to convert it to .(43). which is then reduced with .(44).. In the newer electrolytic process, the roasted ore is then extracted with .(45). and the zinc is plated out on cathodes composed of .(46)..

ZnO 43
coal 44
H_2SO_4 45
aluminum 46

47-50. From the reaction of zinc with sodium hydroxide, it is evident that zinc hydroxide is .(47).. Zinc hydroxide acts as a(n) .(48). in the presence of a strong acid and as a(n) .(49). in the presence of a strong hydroxide. It dissolves in ammonia water by forming the .(50). ion.

amphiprotic 47
hydroxide 48
acid 49
$Zn(NH_3)_4^{++}$ 50

51. Mercury is produced from cinnabar by .(51). this ore.

heating 51

DIRECTIONS: Complete and balance the following equations.

52. $Fe_2O_3 + 3CO \rightarrow$ . . . . . $2Fe(l) + 3CO_2(g)$ 52

53. $CaO + SiO_2 \rightarrow$ . . . . . $CaSiO_3(l)$ 53

54. $3Fe(NO_3)_2 + 4HNO_3 \rightarrow$ . . . . . $3Fe(NO_3)_2 + 2H_2O + NO(g)$ 54

55. $Fe^{+++} + K^+ + Fe(CN)_6^{----} + H_2O \rightarrow$ . . . . . $KFeFe(CN)_6 \cdot H_2O(s)$ 55

56. $Ag^+ + 2CN^- \rightarrow$ . . . . . $Ag(CN)_2^-$ 56

57. $2Au(CN)_2^- + Zn \rightarrow$ . . . . . $Zn(CN)_4^{--} + 2Au(s)$ 57

58. $2ZnS + 3O_2 \rightarrow$ . . . . . $2ZnO + 2SO_2(g)$ 58

59. $Zn + 2NaOH \rightarrow$ . . . . . $Na_2ZnO_2 + H_2(g)$ 59

Exercise 26

NAME. . . . . PERIOD . . . . . DATE. . . . . .

INSTRUCTOR'S COMMENTS . . . . . .

Aluminum and the Metalloids

DIRECTIONS: In the blank space at the right of each statement, write the word or expression that BEST completes the meaning when substituted for the corresponding number.

1-5. Aluminum is .(1). metallic. Characteristics which tend to place it among the metalloids include its appearance with oxygen in negative .(2). ions; its hydroxide is .(3)., its oxide is .(4)., and its hydride is .(5)..

6-7. With the development of the transistor, germanium has become an important .(6). material. It is chemically similar to .(7)..

8-10. The chemistry of tellurium is typically .(8).. It combines with such elements as gold and hydrogen to form .(9).. It combines with oxygen and chlorine and forms compounds in which the oxidation states .(10). are observed.

11-12. Aluminum is extracted by electrolyzing .(11). dissolved in molten .(12)..

13-16. Aluminum has a density of .(13).; it (is, is not) .(14). ductile; it is a (good, poor) .(15). conductor of electricity; it (can, cannot) .(16). be cast.

17-20. Highly polished aluminum soon becomes covered with a thin layer of .(17).. It is an excellent .(18). agent. The Al^{+++} ion is quite .(19). and hydrates .(20). in water solution.

21. Hydrochloric acid (does, does not) .(21). react with aluminum.

22-23. Aluminum reacts in a basic solution and forms the .(22). ion and releases .(23)..

24-26. The formation of aluminum oxide is strongly .(24).. In the thermite reaction, aluminum is used to .(25). oxides of (less, more) .(26). active metals.

27-28. Synthetic rubies and sapphires are made by fusing pure .(27). with an oxyhydrogen torch. An aluminum rod incorporating sapphire .(28). is much stronger than aluminum.

29-32. In the presence of hydrochloric acid, aluminum hydroxide acts as a(n) .(29). forming soluble .(30).. In an excess of sodium hydroxide, aluminum hydroxide acts as a(n) .(31). forming soluble .(32)..

1. distinctly
2. aluminate
3. amphiprotic
4. ionic
5. polymeric
6. semiconductor
7. silicon
8. metalloidal
9. tellurides
10. +2,+4,+6
11. aluminum oxide
12. cryolite
13. 2.7 g/cm^3
14. is
15. good
16. can
17. aluminum oxide
18. reducing
19. small
20. vigorously
21. does
22. aluminate
23. hydrogen
24. exothermic
25. reduce
26. less
27. aluminum oxide
28. "whiskers"
29. base
30. aluminum chloride
31. acid
32. sodium aluminate

33-38. Among the Group III elements, boron has a relatively .(33). ionization energy. It has the .(34). electronegativity in the Group. These characteristics indicate that boron is a(n) .(35). which forms only .(36). bonds with other atoms. As the temperature is raised its electric conductivity .(37).. This behavior is typical of a(n) .(38)..

39-41. Silicon atoms crystallize with a(n) .(39). bond arrangement similar to that of carbon atoms in .(40).. Unlike carbon, silicon forms only .(41). bonds.

42-44. Silicon of high purity is produced by the reduction of silicon dioxide by .(42).. Sand, sandstone, and quartz are commonly called .(43).. Silicon carbide is made by heating .(44). and coke in an electric furnace.

45-46. Silicones are compounds with alternate silicon and .(45). atoms, with .(46). groups attached to the silicon atoms.

47-48. Ordinary glass is made up of sodium and calcium .(47).. The glass made by fusing sand, borax, and aluminum oxide is named .(48)..

49-50. Arsenic and antimony are prepared from their .(49). ores. Antimony is an important constituent of type metal because it causes this alloy to .(50). when it solidifies.

high 33
highest 34
metalloid 35
covalent 36
increases 37
semiconductor 38
tetrahedral 39
diamond 40
single 41
magnesium 42
silica 43
sand 44
oxygen 45
hydrocarbon 46
silicates 47
Pyrex 48
sulfide 49
expand 50

DIRECTIONS: Complete and balance the following equations.

51. $2Al(s) + 6H_3O^+ + 6H_2O \rightarrow$ . . . $2Al(H_2O)_6^{+++} + 3H_2(g)$ . . . 51

52. $2Al(s) + 2NaOH + 2H_2O \rightarrow$ . . . $2NaAlO_2 + 3H_2(g)$ . . . 52

53. $2Al + Fe_2O_3 \rightarrow$ . . . $2Fe + Al_2O_3$ . . . 53

54. $Al(OH)_3(s) + OH^- \rightarrow$ . . . $AlO_2^- + 2H_2O$ . . . 54

55. $Al(OH)_3(s) + 3H_3O^+ \rightarrow$ . . . $Al(H_2O)_6^{+++}$ . . . 55

56. $SiO_2 + 2H_2F_2 \rightarrow$ . . . $SiF_4(g) + 2H_2O$ . . . 56

57. $Na_2CO_3 + SiO_2 \rightarrow$ . . . $Na_2SiO_3 + CO_2(g)$ . . . 57

58. $SiO_2 + 3C \rightarrow$ . . . $SiC + 2CO(g)$ . . . 58

59. $CaCO_3 + SiO_2 \rightarrow$ . . . $CaSiO_3 + CO_2(g)$ . . . 59

60. $Al(H_2O)_6^{+++} + H_2O \rightarrow$ . . . $Al(H_2O)_5(OH)^{++} + H_3O^+$ . . . 60

61. Hall process (a) cathode reaction . . . $4Al^{+++} + 12e^- \rightarrow 4Al$ . . . 61a

(b) two anode reactions . . . $6O^{--} \rightarrow 3O_2 + 12e^-$ . . . 61b

. . . $3C + 3O_2 \rightarrow 3CO_2(g)$ . . . 61b

62. $HAsO_2 + 3Zn + 6H^+(aq) \rightarrow$ . . . $AsH_3(g) + 3Zn^{++} + 2H_2O$ . . . 62

Exercise 27

NAME. . . . . PERIOD . . . . . DATE . . . . . .
INSTRUCTOR'S COMMENTS . . . . . .

Nitrogen and its Compounds

DIRECTIONS: In the parenthesis at the right of each word or expression in the second column, write the letter of the expression in the first column which is most closely related.

a. strong triple covalent bond	natural combined nitrogen	(k)	1
b. test for nitrogen	Rutherford	(e)	2
c. brown layer test	plastics	(h)	3
d. moist blue litmus turns red	nitrogen dioxide	(d)	4
e. first separation of nitrogen from air	magnesium nitride	(b)	5
f. lightning discharge through air	nitrogen monoxide	(f)	6
g. cleaning agent	ammonium ion	(i)	7
h. nitrocellulose	ammonia-water solution	(g)	8
i. tetrahedral structure	inactivity of nitrogen	(a)	9
j. nitrobenzene	nitrate ion	(c)	10
k. proteins			
l. hydrazine			

DIRECTIONS: Complete the following statements which refer to the diagram at the left, forming accurate and complete sentences.

11. In preparing ammonia in the laboratory, a nitrogen-containing compound which might be placed in the test tube is . . . ammonium chloride 11

12. Mixed with this substance should be . . . calcium hydroxide 12

13. Ammonia is collected in this manner because it is . . . soluble in water and less dense than air 13

14. When the test tube is filled with ammonia, the wet litmus test strip will change to . . . blue 14

15. The solid remaining in the test tube after the reaction is . . . calcium chloride 15

DIRECTIONS: In the blank space at the right of each statement, write the word or expression which BEST completes the meaning when substituted for the corresponding number.

16. Traces of ammonia are always found in the air because it is a decomposition product of .(16).. . . . proteins 16

17-20. The process of producing ammonia by direct combination of nitrogen and hydrogen is called the .(17). process. This process requires .(18). pressure, .(19). temperature, and the presence of a mixture of iron and the oxides of potassium and aluminum as a(n) .(20)..

17. . . . Haber 17
18. . . . high 18
19. . . . high (moderately) 19
20. . . . catalyst 20

21-24. The electron-dot formula for ammonia is .(21).. Its pyramidal structure is evidence of .(22). hybridization in the molecule. Its unusually high boiling point is explained by the formation of .(23). between liquid ammonia molecules. Because the hydrogen atoms are not symmetrically bonded to the nitrogen atom, the ammonia molecules are .(24)..

21. . . . H:N:H (H below N) 21
22. . . . sp^3 22
23. . . . hydrogen bonds 23
24. . . . polar 24

25-27. On a farm, ammonia may be used as a(n) .(25).. In the home, it may be used as a(n) .(26).. In frozen food locker plants, it is used as a(n) .(27)..

25 fertilizer
26 cleaning agent
27 refrigerant

28. The nitrogen and hydrogen compound used as a rocket fuel is .(28)..

28 hydrazine

DIRECTIONS: Write the answers to the following in the spaces provided. Where appropriate, make complete statements.

29. Why are the Haber process and the Ostwald process important and related processes?
29 They make possible the production of nitric acid and nitrates from readily available raw materials. Ammonia produced by the Haber process is converted to nitric acid by the Ostwald process.

30. Why does nitric acid exhibit both the properties of acids and the properties of oxidizing agents?
30 The hydronium ions give it its acid properties, while the nitrogen in the nitrate ions act as an oxidizing agent.

31. What factors determine the nitrogen compounds produced by the action between metals and nitric acid?
31 The concentration and temperature of the acid and the metal which reacts.

32. Why can hydrogen be replaced in very dilute nitric acid by metals like sodium, calcium, and magnesium?
32 These are very active metals and reduce the hydrogen in preference to the nitrogen.

33. With which metals does nitric acid *not* react? Why?
33 It does not react with gold or platinum because of their inactivity.

34. Describe the test for a nitrate.
34 Slowly pour a few milliliters of concentrated sulfuric acid down the inclined wall of a test tube containing a mixture of the unknown and iron(II) sulfate solution. If a brown layer forms at the liquid junction, the unknown contained a nitrate.

35. List four important uses for nitric acid.
35 For making fertilizers, dyes, plastics, and explosives.

DIRECTIONS: Complete and balance the following equations.

36. $NH_4Cl(aq) + NaNO_2(aq) \rightarrow$ 36 $NH_4NO_2(aq) + NaCl(aq) \searrow N_2(g) + 2H_2O(l)$

37. $CaC_2 + N_2 \rightarrow$ 37 $CaCN_2 + C$

38. $NaOH(s) + NH_4Cl(s) \rightarrow$ 38 $NaCl(s) + NH_3(g) + H_2O(g)$

39. $NH_3 + H_2O \rightleftarrows$ 39 $NH_4^+ + OH^-$

40. $N_2(g) + 3H_2(g) \rightleftarrows$ 40 $2NH_3(g)$

41. $NaNO_3(s) + H_2SO_4(aq) \rightarrow$ 41 $NaHSO_4(aq) + HNO_3(g)$

42. $4NH_3 + 5O_2 \rightarrow$ 42 $4NO + 6H_2O$

43. $2NO + O_2 \rightarrow$ 43 $2NO_2$

44. $3NO_2(g) + H_2O(l) \rightarrow$ 44 $2HNO_3(aq) + NO(g)$

45. $3Cu(s) + 8H^+(aq) + 2NO_3^-(aq) \rightarrow$ 45 $3Cu^{++}(aq) + 2NO(g) + 4H_2O(l)$

Exercise 28

NAME. . . . . PERIOD . . . . . DATE . . . . . .
INSTRUCTOR'S COMMENTS . . . . . .

Sulfur and its Compounds

DIRECTIONS: In the parentheses at the right of each word or expression in the second column write the letter of the expression in the first column which is *most closely* related.

a. $Pb(C_2H_3O_2)_2$	a white paint pigment	(i)	1
b. SO_3	roasting	(c)	2
c. FeS_2	test for hydrogen sulfide	(a)	3
d. CS_2	preparation of paper pulp	(h)	4
e. H_2SO_3	a weak acid	(e)	5
f. $BaCl_2$	test for a sulfate	(f)	6
g. SO_2	contact catalyst	(j)	7
h. $Ca(HSO_3)_2$	drying gases	(k)	8
i. $Ca_3(PO_4)_2$	fruit preservative	(g)	9
j. V_2O_5	rubber solvent	(d)	10
k. H_2SO_4			
l. $ZnSO_4$			

DIRECTIONS: Write the answers to the following in the spaces provided. Make complete statements where appropriate.

11. In what forms does sulfur occur in nature? . . . . . As the free element or combined with other elements in sulfides and sulfates. 11

12. What is superheated water? Water that is heated under pressure to above its normal boiling point. 12

13. Why is superheated water used in the Frasch process? . . . . . The superheated water (170° C) melts the sulfur (melting point 114.5° C). 13

14. Why does λ-sulfur flow easily? The rounded S_8 molecules roll over one another easily. 14

15. Why is μ-sulfur very viscous? The chains of sulfur atoms become tangled. 15

16. Why does μ-sulfur become darker in color? . . . . . Because the increasing number of free electrons absorbs a greater amount of light. 16

17. What are the common oxidation numbers of sulfur respectively represented in the compounds SO_3, SO_2, and H_2S? +6; +4; −2. 17

18. To what element is sulfur similar in the manner in which it combines with other elements? Oxygen 18

19. What method is used for collecting either hydrogen sulfide or sulfur dioxide in the laboratory? Upward displacement of air. 19

20. Why are these gases collected by this method? . . . . . They are denser than air and soluble in water. H_2S is moderately soluble and SO_2 is very soluble. 20

21. What property of hydrogen sulfide is shown by its reaction with hydrogen peroxide? . . . . . Hydrogen sulfide is a reducing agent. 21

22. How is the sulfide ion concentration in saturated hydrogen sulfide solutions related to the hydronium ion concentration? The sulfide ion concentration varies inversely with the square of the hydronium ion concentration. 22

23. If a yellow precipitate is produced when hydrogen sulfide is bubbled through an unknown salt solution, what metal(s) may have been present? As arsenic(III), arsenic(V); tin(IV); cadmium. 23

24. At what pH are the sulfides of (a) arsenic through lead and (b) zinc through manganese precipitated? (a) pH = 1; (b) pH = 9. 24

25. Why are sulfide ores roasted? To convert the sulfur of the ore to sulfur dioxide (and the metal to a metallic oxide) as the first step in extracting the metal from the ore. 25

26. What is the role of the reagents (a) copper and (b) hot, concentrated sulfuric acid in the laboratory method for preparing sulfur dioxide? (a) Reducing agent; (b) oxidizing agent 26

27. What are the anhydrides respectively of (a) sulfurous acid and (b) sulfuric acid? (a) Sulfur dioxide; (b) sulfur trioxide. 27

28. How, according to current belief, does sulfurous acid bleach? It converts the colored compounds to colorless sulfites. 28

29. How is sulfur dioxide converted to sulfur trioxide? By heating it with air in the presence of a catalyst such as vanadium pentoxide. 29

30. How is sulfur trioxide converted to sulfuric acid in the contact process? It is dissolved in approximately 98% sulfuric acid forming pyrosulfuric acid, which, when diluted with water yields sulfuric acid. 30

31. How is sulfuric acid diluted safely? Why is this precaution necessary? The acid is added to the water (you must never add water to sulfuric acid) since a great deal of heat is evolved because of the formation of hydrates $H_2SO_4 \cdot H_2O$ and $H_2SO_4 \cdot 2H_2O$. 31

32. Why does dilute sulfuric acid react more readily with metals above hydrogen (in the series of oxidizing and reducing agents) than cold, concentrated acid does? Because it is more highly ionized. 32

33. What kind of sulfuric acid acts as a vigorous oxidizing agent? Hot concentrated sulfuric acid is an oxidizing agent. 33

34. Why is sulfuric acid used in the manufacture of nitroglycerin? It acts as a dehydrating agent, absorbing water as fast as it is formed and thereby maintaining the rate of reaction. 34

35. Why is hydrochloric acid used in the test for a sulfate? The hydrochloric acid prevents the formation of such interfering precipitates as barium oxalate, barium carbonate, barium sulfite, or barium phosphate which might be confused with the barium sulfate precipitate. 35

DIRECTIONS: Complete and balance the following equations.

36. $S_8(s) + 8O_2(g) \rightarrow$ $8SO_2(g)$ 36

37. $FeS(s) + 2H^+(aq) \rightarrow$ $Fe^{++}(aq) + H_2S(g)$ 37

38. $2H_2S(g) + 3O_2(g) \rightarrow$ $2SO_2(g) + 2H_2O(g)$ 38

39. $H_2S + H_2O \rightleftarrows$ $H_3O^+ + HS^-$ 39

40. $HS^- + H_2O \rightleftarrows$ ..... $H_3O^+ + S^{--}$ 40

41. $2ZnS(s) + 3O_2(g) \rightarrow$ ..... $2ZnO(s) + 2SO_2(g)$ 41

42. $Cu(s) + 2H_2SO_4(hot) \rightarrow$ ..... $CuSO_4(aq) + 2H_2O(l) + SO_2(g)$ 42

43. $Na_2SO_3(aq) + H_2SO_4(aq) \rightleftarrows$ ..... $Na_2SO_4(aq) + H_2O(l) + SO_2(g)$ 43

44. $H_2O(l) + SO_2(aq) \rightleftarrows$ ..... $H_2SO_3(aq)$ 44

45. $H_2SO_3 + H_2O \rightleftarrows$ ..... $H_3O^+ + HSO_3^-$ 45

46. $SO_3 + H_2O \rightarrow$ ..... $H_2SO_4(l)$ 46

47. $2SO_2(g) + O_2(g) \rightleftarrows$ ..... $2SO_3(g)$ 47

48. $H_2SO_4 + H_2O \rightleftarrows$ ..... $H_3O^+ + HSO_4^-$ 48

49. $HSO_4^- + H_2O \rightleftarrows$ ..... $H_3O^+ + SO_4^{--}$ 49

50. $4Zn(s) + 5H_2SO_4(hot) \rightarrow$ ..... $4ZnSO_4(aq) + H_2S(g) + 4H_2O(l)$ 50

51. $C_{12}H_{22}O_{11} + 11H_2SO_4 \rightarrow$ ..... $12C + 11H_2SO_4 \cdot H_2O$ 51

52. $C_3H_5(OH)_3 + 3HNO_3 \rightarrow$ ..... $C_3H_5(NO_3)_3 + 3H_2O$ 52

53. $Ba^{++}(aq) + SO_4^{--}(aq) \rightarrow$ ..... $BaSO_4(s)$ 53

DIRECTIONS: Solve the following problems. Show calculations in the space provided and enter your answer on the dotted line at the bottom of the space.

54. The K obtained by multiplying the ionization constants for the two stages of ionization of H_2S is 6.8×10^{-23}. Saturated solutions of H_2S in water are approximately 0.1 M with respect to H_2S. Calculate the sulfide ion concentration in a saturated solution of H_2S in which the pH = 3.

Answer: ..... $6.8 \times 10^{-18}\ M$ 54

55. The K_{sp} for CoS is 8.7×10^{-23}. Calculate the concentration of sulfide ion which must be exceeded in order for CoS to precipitate in a solution in which the Co^{++} concentration is 0.001 M .....

..... 8.7×10^{-20} 55

Exercise 29

NAME . . . . . PERIOD . . . . . DATE . . . . . .

INSTRUCTOR'S COMMENTS . . . . . .

Halogen Family

DIRECTIONS: In the blank space at the right of each statement, write the word or expression that BEST completes the meaning when substituted for the corresponding number.

1-5. The Halogen Family consists of the (metallic, nonmetallic) .(1). elements: fluorine, chlorine, bromine, iodine, and astatine. The atoms of each of these elements have .(2). outershell electrons and commonly exhibit an oxidation number of .(3).. The halogens are (active, inactive, inert) .(4). elements which are (always, sometimes, rarely, never) .(5). found free in nature.

nonmetallic 1

seven 2

-1 3

active 4

rarely 5

6-10. Within the Halogen Family, the electronegativity .(6). with increasing atomic diameter, so that .(7). is the most electronegative halogen. The melting point, boiling point, and density of the members of the Halogen Family .(8). with increasing atomic number. The compounds of the halogens and metals are formed by .(9). bonding. The hydrogen halides are formed by .(10). bonding.

decreases 6

fluorine 7

increase 8

ionic 9

covalent 10

11-17. The halogens are prepared commercially by different processes: fluorine by the .(11). of a mixture of .(12). and hydrogen fluoride; chlorine by the electrolysis of .(13).; bromine by .(14). from .(15). by chlorine; iodine by displacement from .(16). by .(17)..

electrolysis 11

potassium fluoride 12

sodium chloride 13

displacement 14

brine 15

brine 16

chlorine 17

18-21. The halogens are colorful elements. Fluorine is a(n) .(18). gas; chlorine, a(n) .(19). gas; bromine, a(n) .(20). liquid, and iodine, a(n) .(21). solid.

pale-yellow 18

greenish-yellow 19

reddish-brown 20

grayish-black 21

22-24. Hydrogen fluoride may be prepared by treating .(22). with sulfuric acid. Hydrofluoric acid is used as a(n) .(23). in the manufacture of high-octane gasoline, and in the production of synthetic .(24). for aluminum production.

calcium fluoride 22

catalyst 23

cryolite 24

25-30. Hydrogen chloride may be prepared commercially by heating .(25). with .(26).. Liquid hydrogen chloride consists of .(27). molecules and therefore .(28). conduct electricity. Hydrochloric acid is used for .(29). metals before they are galvanized or plated with other metals. It is a component of a secretion which promotes the body process of .(30)..

sodium chloride 25

sulfuric acid 26

polar covalent 27

does not 28

cleaning 29

digestion 30

DIRECTIONS: Write the answers to the following in the spaces provided. Make complete statements where appropriate.

31. What is the formula, chemical name, and commercial name of the fluorine compound used as a refrigerant? CCl_2F_2, dichlorodifluoromethane, a Freon 31

32. What type of bonding occurs between HF molecules to produce H_2F_2 and H_3F_3 molecules? hydrogen bonding 32

33. Why is hydrofluoric acid a weak acid?
Only a few of the H_2F_2 molecules present in the acid solution ionize. 33

34. What are the formulas of the fluorides of xenon that are white solids at ordinary temperatures? XeF_2, XeF_4, XeF_6.
What are the products of their reaction with hydrogen?
Elementary xenon and hydrogen fluoride. 34

35. How do the compounds of Question 34 react with water?
XeF_2 produces xenon, oxygen, and hydrogen fluoride. The other two yield xenon trioxide. 35

36. Why is carbon liberated in the reaction between turpentine and chlorine?
Chlorine has such a great attraction for hydrogen that it takes the hydrogen from the turpentine and liberates carbon. 36

37. How does a water solution of chlorine bleach a dye?
The hypochlorous acid formed liberates oxygen which converts an oxidizable dye to a colorless compound. 37

38. What are three uses for bromine? As a sedative in medicine, making photographic film and plates, in making ethylene bromide which is used to increase the efficiency of lead tetraethyl in antiknock gasoline, etc. 38

39. What is the purpose of the carbon tetrachloride in the tests for bromide and iodide ions?
It acts as a solvent for the liberated bromine and iodine. 39

40. Why is iodine more soluble in solutions of iodides than it is in water?
Because it forms the complex I_3^- ion in solutions of iodides. 40

DIRECTIONS: Write complete and balanced equations for the following reactions.

41. $CaF_2(s) + H_2SO_4(l) \rightarrow$ $CaSO_4(s) + 2HF(g)$ 41

42. $H_2O(l) + H_2F_2(aq) \rightleftarrows$ $H_3O^+(aq) + HF_2^-(aq)$ 42

43. $2NaCl(aq) + 2H_2O(l) \xrightarrow{\text{(elect)}}$ $2NaOH(aq) + H_2(g) + Cl_2(g)$ 43

44. $MnO_2(s) + 4HCl(aq) \rightarrow$ $MnCl_2(aq) + 2H_2O(l) + Cl_2(g)$ 44

45. $2Sb(s) + 3Cl_2(g) \rightarrow$ $2SbCl_3(s)$ 45

46. $NaCl(s) + H_2SO_4(aq) \rightarrow$ $NaHSO_4(aq) + HCl(g)$ 46

47. $Ag^+Cl^-(s) + 2NH_3(aq) \rightarrow$ $Ag(NH_3)_2^+(aq) + Cl^-(aq)$ 47

48. $Ag(NH_3)_2^+(aq) + Cl^-(aq) + 2H^+(aq) + 2NO_3^-(aq) \rightarrow$ $Ag^+Cl^-(s) + 2NH_4^+(aq) + 2NO_3^-(aq)$ 48

49. $2Br^-(aq) + Cl_2(g) \rightarrow$ $2Cl^-(aq) + Br_2(l)$ 49

50. $2NaI(s) + MnO_2(s) + 2H_2SO_4(aq) \rightarrow$ $Na_2SO_4(aq) + MnSO_4(aq) + 2H_2O(l) + I_2(g)$ 50

Exercise 30

NAME . . . . . PERIOD . . . . . DATE . . . . .

INSTRUCTOR'S COMMENTS . . . . . .

Radioactivity

DIRECTIONS: In the blank space at the right of each definition, write the name of the term defined.

1. The property of substances which give off invisible rays that affect an unexposed photographic plate in the same way as light. radioactivity 1

2. A device used to detect radiation by the ionization of low-pressure gas in a tube. Geiger counter 2

3. The glow which radium compounds produce when mixed with certain compounds like zinc sulfide. fluorescence 3

4. The length of time required for the decay of one-half of a given number of atoms of a radioactive nuclide. half-life 4

5. Helium nucleus emitted from the nucleus of a radioactive nuclide. alpha particle 5

6. Electron emitted from the nucleus of a radioactive nuclide. beta particle 6

7. High-energy electromagnetic waves emitted from the nucleus of a radioactive nuclide. gamma rays 7

8. The heaviest nuclide of a series of related radioactive nuclides. parent nuclide 8

9. A reaction in which the nucleus of an atom undergoes a change in the number of protons, and consequently in its identity. transmutation 9

10. The difference between the measured mass of a nucleus and the sum of the masses of its constituent particles. nuclear mass defect 10

11. The energy released when a nucleus is formed from its component particles. binding energy 11

12. The emission of an alpha particle or a beta particle and gamma rays from a nucleus producing a slightly lighter, more stable nucleus. radioactive decay 12

13. The emission of a proton or neutron from a nucleus as a result of bombarding the nucleus with alpha particles, protons, deuterons, neutrons, etc. nuclear disintegration 13

14. The splitting of a very heavy nucleus into medium-weight nuclei. fission 14

15. The combination of light-weight nuclei to form heavier, more stable nuclei. fusion 15

16. A particle with zero atomic number and a mass number of one. neutron 16

17. A device which accelerates electrons rather than positively charged particles as does the synchrotron. betatron 17

18. A device in which particles travel in a straight line through many stages of small potential difference which act to accelerate the particles. linear accelerator 18

19. Elements with more than 92 protons in their nuclei. transuranium elements 19

20. A radioactive isotope that is used to determine the course of chemical reactions. radioactive tracer 20

21. A reaction in which the material or energy which start the reaction is also one of the products. chain reaction 21

22. A device in which the controlled fission of radioactive material produces new radioactive substances and energy. nuclear reactor 22

23. A material which slows down fast neutrons and makes them more effective for producing nuclear changes. moderator 23

24. A neutron-absorbing shaft inserted into a nuclear fuel lattice to limit the number of free neutrons. control rod 24

25. The quantity of uranium necessary to maintain a chain reaction. critical size 25

DIRECTIONS: In the parentheses at the right of each name or pair of names in the second column, write the letter of the expression in the first column which is most closely related.

a. predicted relationship between matter and energy
b. produced first artificial radioactive nuclides
c. invented the cyclotron
d. built the first nuclear reactor
e. discovered neutrons
f. discovered polonium
g. produced first nuclear disintegration
h. isolated uranium
i. constructed a counter tube
j. verified Einstein's equation
k. studied fluorescence in minerals

Becquerel	(i) 26
Chadwick	(e) 27
Cockcroft and Walton	(j) 28
Pierre and Marie Curie	(f) 29
Geiger	(i) 30
Einstein	(a) 31
Frederic and Irene Joliot	(b) 32
Lawrence	(c) 33
Rutherford	(g) 34

DIRECTIONS: In Questions 35-40, complete the nuclear equations which represent several steps in the disintegration of a $^{235}_{92}U$ nuclide. Use the product of one reaction as the reactant in the next one.

35. $^{235}_{92}U \rightarrow {}^{4}_{2}He + {}^{231}_{90}Th$ .....

36. $^{231}_{90}Th$ ..... $\rightarrow {}^{0}_{-1}e + {}^{231}_{91}Pa$ .....

37. $^{231}_{91}Pa$ ..... $\rightarrow {}^{4}_{2}He + {}^{227}_{89}Ac$ .....

38. $^{237}_{89}Ac$ ..... $\rightarrow {}^{0}_{-1}e + {}^{237}_{90}Th$ .....

39. $^{227}_{90}Th$ ..... $\rightarrow {}^{4}_{2}He + {}^{223}_{88}Ra$ .....

40. $^{223}_{88}Ra$ ..... $\rightarrow {}^{4}_{2}He + {}^{219}_{86}Rn$ .....

DIRECTIONS: In questions 41-46, complete the equations by supplying the missing information. Use the tables in the Appendix of MODERN CHEMISTRY for names, symbols, and atomic numbers of the elements.

41. $^{14}_{7}N + {}^{4}_{2}He \rightarrow {}^{1}_{1}H + {}^{17}_{8}O$ .....

42. $^{7}_{3}Li + {}^{1}_{1}H \rightarrow {}^{4}_{2}He + {}^{4}_{2}He$ .....

43. $^{9}_{4}Be + {}^{4}_{2}He \rightarrow {}^{12}_{6}C + {}^{1}_{0}n$ .....

44. $^{239}_{92}U \rightarrow {}^{0}_{-1}e + {}^{239}_{93}Np$ .....

45. $^{32}_{16}S + {}^{1}_{0}n \rightarrow {}^{1}_{1}H + {}^{32}_{15}P$ .....

46. $^{239}_{94}Pu + 2\,{}^{1}_{0}n \rightarrow {}^{0}_{-1}e + {}^{241}_{95}Am$ .....

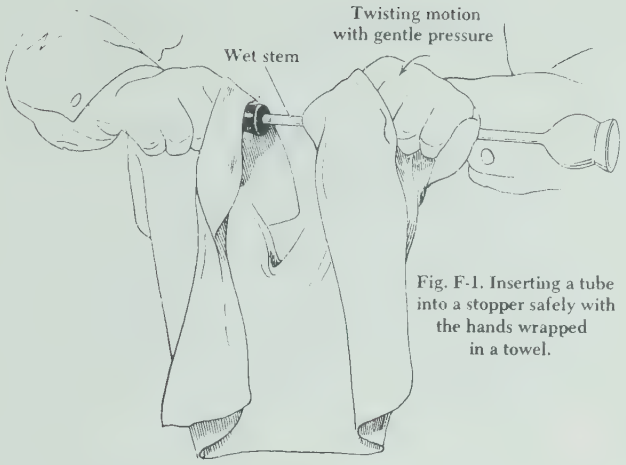


Fig. F-1. Inserting a tube into a stopper safely with the hands wrapped in a towel.

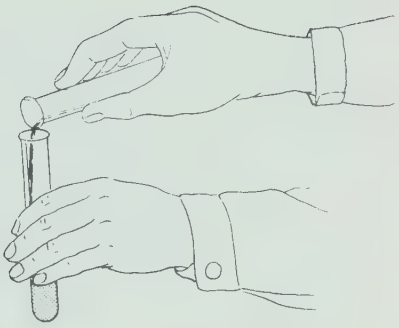


Fig. F-2. Transferring a liquid safely at arm's length.

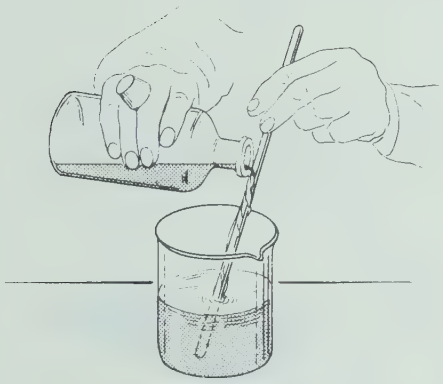


Fig. F-3. Pouring an acid into water safely.

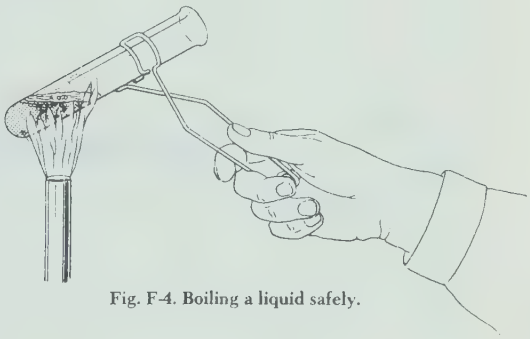
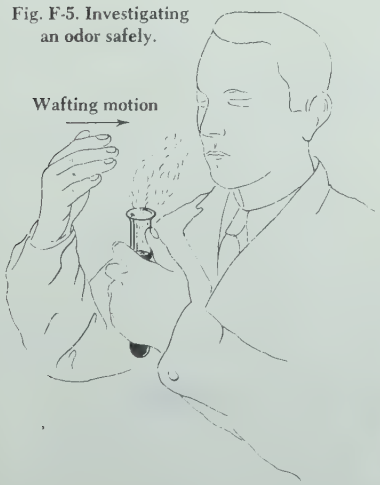


Fig. F-4. Boiling a liquid safely.

Fig. F-5. Investigating an odor safely.



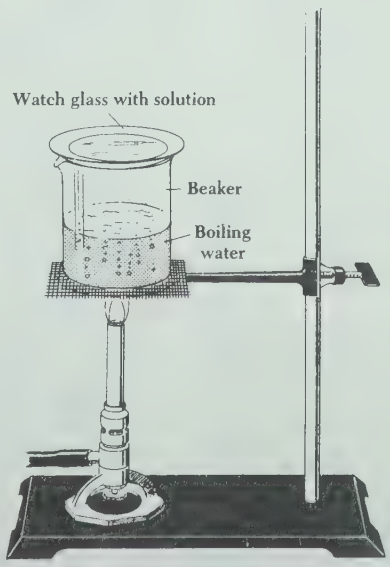


Fig. F-6. Evaporating over a water bath.

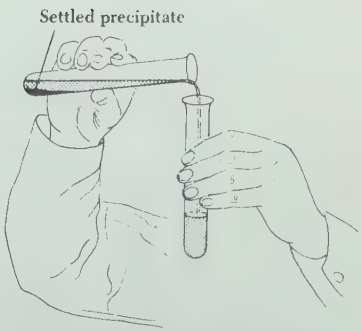


Fig. F-7. Decanting a supernatant liquid without a stirring rod.

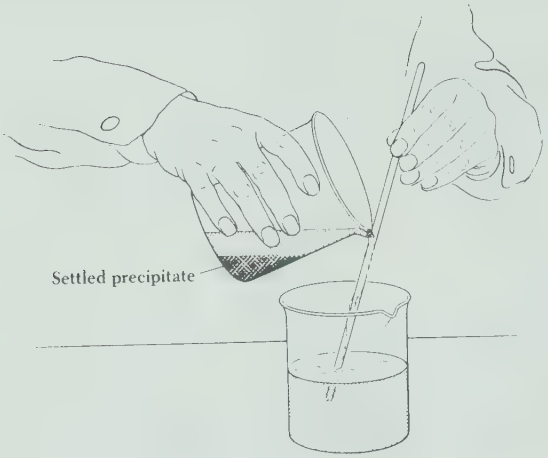


Fig. F-8. Decanting a supernatant liquid using a stirring rod.

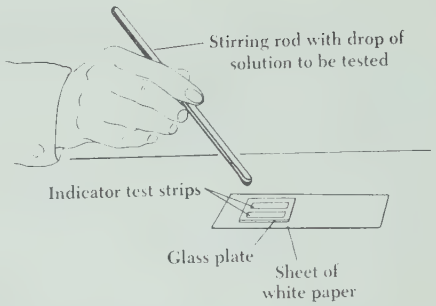


Fig. F-9. Testing a solution with indicator paper.

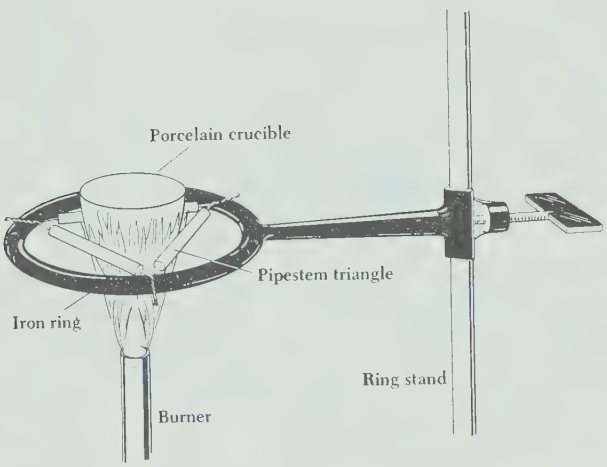


Fig. F-10. Heating a substance in a crucible.

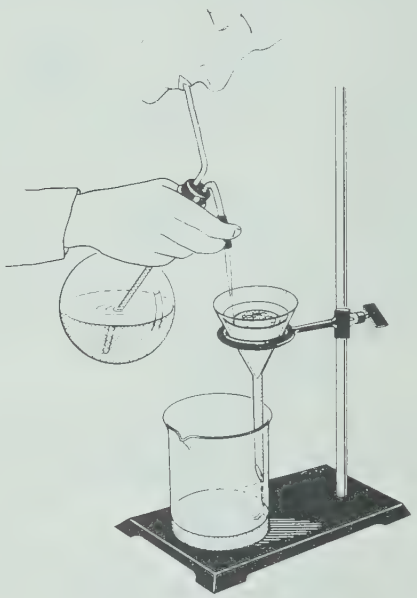


Fig. F-11. Washing a precipitate using a wash bottle.

Fig. F-12. Using a wash bottle.

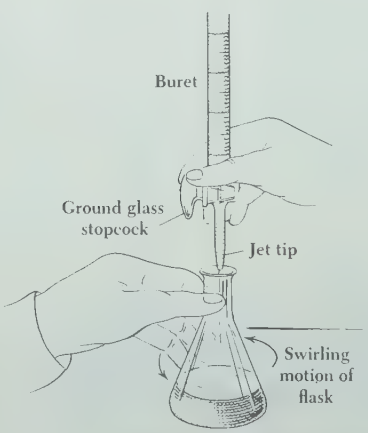


Fig. F-13. Titrating a solution using a buret.

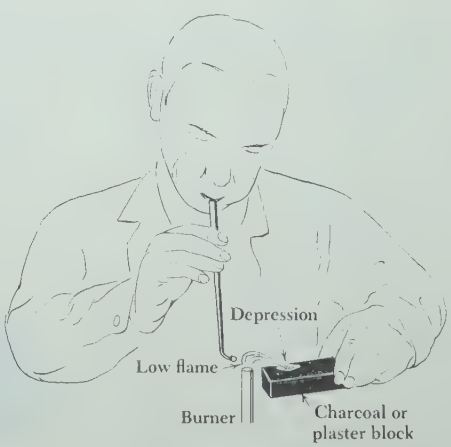


Fig. F-14. Using a blowpipe.

Experiment 1

NAME . . . . . PERIOD . . . . . DATE . . . . . .
INSTRUCTOR'S COMMENTS . . . . . .

Laboratory Procedures

PURPOSE: To become acquainted with laboratory apparatus and laboratory techniques.

APPARATUS: Asbestos square; beakers, 250 ml, 400 ml; balance, platform, buret; burner and tubing; calorimeter (plastic cups and lid); clamp, buret; dish, evaporating; file, triangular; flasks, Florence, 250 ml, 500 ml; forceps; funnel; funnel tube; graduated cylinder; pinch clamp; pipet, 25 ml; ring, iron; ring stand; rubber stoppers, 2-hole No. 4, 2-hole No. 5; test tube; thermometer; wing top; wire gauze.

MATERIAL: Copper wire, 18 gauge; filter paper, glass rod, 6 mm; glass tubing, 6 mm; potassium permanganate; sand; sodium chloride.

INTRODUCTION: The best way to become familiar with chemical apparatus is to actually handle the pieces yourself in the laboratory. In this experiment, you will learn how to adjust the bas burner, manipulate glass tubing, make weighings, handle solids, measure liquids, filter a mixture, and assemble apparatus. Several useful manipulative techniques are illustrated on pages 95-96 under the heading, Techniques and Safety Sketches. Reference to them will be made in this experiment and in several later experiments.

SUGGESTION: If you use plastic wash bottles you may wish to omit this procedure. However it does provide a project for those students who do the glass bending, cutting, and fire polishing. Wash bottles, once made, may be stored in a wall case where they are readily accessible for use. In multiple-section laboratories the instructor may wish to store one set of wash bottles with solid stoppers in the wall case and have each student retain his own stopper assembly in his locker. Other subassemblies which may be stored in sets or in individual student lockers (Fig. 1-14) are time savers in subsequent experiments. Two full laboratory periods are needed for this experiment.

PROCEDURE: 1. *The burner.* The Bunsen or Tirrell burner is commonly used in laboratory heating operations. While the details of construction vary among burners, each has a gas inlet located in the base, a vertical tube or barrel in which the gas is mixed with air, and adjustable openings or ports in the base of the barrel to introduce air into the gas stream. The burner may have an adjustable needle valve to regulate the supply of gas, or the gas supply may be regulated simply by adjusting the valve on the supply line. The burner is always turned off at the gas valve, never at the needle valve.

Take your burner apart and examine it. Compare the parts with the drawing in Fig. 1-1. Put the parts together again and note particularly how you can control the amount of air admitted near the base of the burner. In lighting the burner, partially close the ports at the base of the barrel, turn the gas full on, and hold the lighted match about 5 cm above the top of the burner. The gas may then be regulated until the flame has the desired height. If a very low flame is needed, the ports should be kept partly closed when the gas pressure is reduced. Otherwise the flame may "strike back" and burn inside the base of the barrel. If this happens, turn off the gas, decrease the amount of air admitted, and relight.

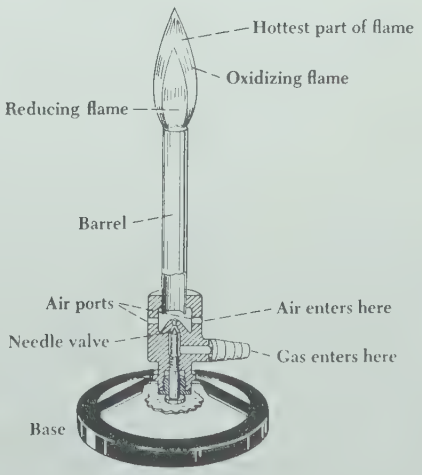


Fig. 1-1

Close the holes at the base. What is the result? How would you describe the flame? Using the forceps, hold an evaporating dish in the tip of the flame. Result? Suggest a possible explanation. Such a flame is seldom used in the laboratory. Why? For laboratory work, you should adjust the burner so that the flame will be free of yellow color, and also free from the "roaring" sound caused by admitting too much air.

Regulate the flow of gas to give a flame extending roughly 8 cm above the barrel. Now adjust the supply of air until you have a quiet, steady flame with a sharply defined inner cone. This adjustment gives the highest temperature possible with your burner. Using the forceps, insert a 10-cm piece of copper wire into the flame just above the barrel. Lift the wire slowly up through the flame. Where is the hottest portion of the flame located? Hold the wire in this part of the flame for a few seconds. Result? The melting point of copper is 1083° C.

2. *Glass Manipulations. a. Cutting tubing.* Secure several pieces of glass tubing from the scrap supply to use for practice. At the point to be cut, make a deep scratch at right angles by pushing the edge of a triangular file away from you with *one* firm, steady stroke. Grasp the tubing with both hands, with your thumbs meeting opposite the scratch, and break it by pulling the ends toward you. Repeat until you have mastered this operation.

b. Fire polishing. The edges of the tubes you have just cut are very sharp, and they would cut a rubber stopper or rubber tubing if you tried to use the tube with either one. *Fire polish* the end of the tube by holding it in the burner flame, rolling it between the fingers to heat it uniformly. Recall the location of the hottest part of the burner flame as determined in Part 1. As the flame above the tubing yellows, the glass softens, and its sharp edges become rounded. **CAUTION:** *If the end of the tube is held in the flame too long, the size of the opening will be reduced.* Place hot glass tubing on an asbestos square to cool.

c. Bending tubing. Place a wing top on the barrel of your burner, in order to give a broad, flat flame. Select pieces of glass tubing from the scrap supply for practice. Hold the tubing in the flame so that it will be heated for about 5 cm of its length at the place where it is to be bent. Rotate the tubing constantly to heat all sides uniformly. When the flame above the tubing becomes an intense yellow, the glass will be soft enough to bend easily. Remove it from the flame, and bend it to make a smooth 90° bend similar to that shown in *b* of Fig. 1-2. Fire polish the ends and place the tube on the asbestos square to cool. Be sure that you have both arms of the bent tube in the same plane.

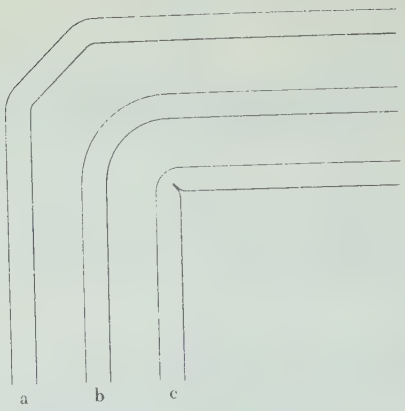


Fig. 1-2

Repeat the procedure with additional pieces of scrap glass tubing until you are able to form a satisfactory bend. Now secure a length of tubing, 6 mm in diameter, and cut, bend, and fire polish 90° glass bends as follows: one 8 × 8 cm, one 8 × 15 cm, and one 8 × 30 cm. Secure a 15-cm length of 6-mm glass rod and fire polish both ends to form a *stirring rod*. Retain all 4 pieces in your locker for future use.

d. Drawing glass tubing. Again secure a few picces of scrap tubing to use for practice. Remove the wing top from your burner and hold the middle region of a piece of tubing in the flame as you heat it a little hotter than necessary for bending. Allow the walls to thicken somewhat. Remove it from the flame and pull the two ends apart until the softened region is as small as desired, making the pull straight and with even tension. To make a *jet tip*, cut the constricted portion to leave a jet tip about 6 or 8 cm in length. Fire polish both ends, being careful not to seal the jet end. Using 6-mm glass tubing, make 3 well-formed jet tips to retain in your locker. To make a *closed tube*, cut off the constricted part as before, and then hold the jet end in the flame until it is melted shut.

3. *The balance.* For most purposes in general chemistry, the moderately accurate weighings possible on the ordinary platform balance are satisfactory. Such a balance is shown in Fig. 1-3. It is sensitive to 0.1 g.

More precise work in the laboratory may require a balance of greater sensitivity. Such a balance is shown in Fig. 1-4. It is sensitive to 0.01 g and is very useful in certain quantitative experiments. In order to carry out

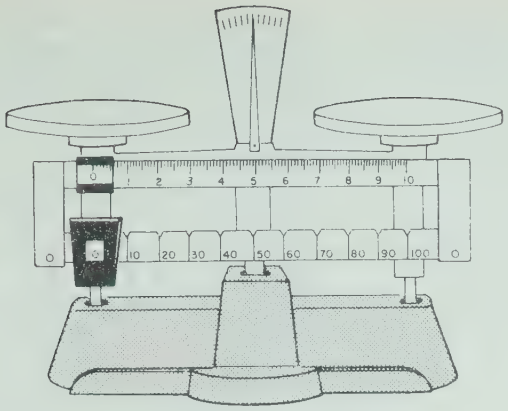


Fig. 1-3

a weighing successfully, you should learn the following steps.

a. Determine the rest point. Inspect your platform balance to see that both pans are clean and that the pointer swings, or oscillates, freely over the range of the attached scale with the slider (slide weight) at zero. *The rest point is at the center of the range over which the pointer swings, determined to the nearest half-scale division.* It is not necessarily at the center of the attached scale. The rest point can be found only if the pointer is oscillating within the range of the fixed scale. A typical rest point determination is illustrated in Fig. 1-5. If a weighing paper is to be used in making a weighing, place a paper square on each platform before determining the rest point. If the rest point is more than 3 scale divisions from the center of the scale, or if the pointer sticks in any part of its oscillation, call this to the attention of your instructor. Do not attempt to make independent adjustments on your balance.

b. Balance the substance and known masses. Place a small square of glazed paper on each platform of the balance (discs of filter paper will do if glazed paper is not available). Determine the rest point. Examine the balance carefully to determine which pan should be used to support the material whose mass is to be measured. Put an appropriate amount of dry sand on this pan. Move the slider on the 0-100 g beam into the slot, which you judge, indicates the upper portion of the 10-g range within which the mass of the sand falls. For example, if use of this slider indicates that the mass of the sand is more than 10 g but less than 20 g, the slider is first placed in the 20-g position and then moved back to the lower part of the range, that is, the 10-g position. Now move the slider on the 0-10-g beam until the pointer oscillations remain within the range of the fixed scale. Adjust this slider so that the midpoint of the oscillation corresponds to the rest point of the balance. Record in your notebook the mass of the sand which is determined by the sum of the slider readings of both beams. For example, if the reading on the 0-100-g beam is 50 g and that on the 0-10 g beam is 4.3 g, the total mass which you are measuring is 54.3 g. For a balance accurate to 0.1 g, make sure that you record to one place to the right of the decimal point. Always label your record and retain it in your laboratory notes.

Sometimes only a close approximation is needed. Suppose you wish to use about 10 grams of a solid. Place a sheet of glazed paper on each pan, and move the slider to the extreme right, at the 10-g mark. Using a horn spoon or spatula, add the solid to the left pan until an approximate balance

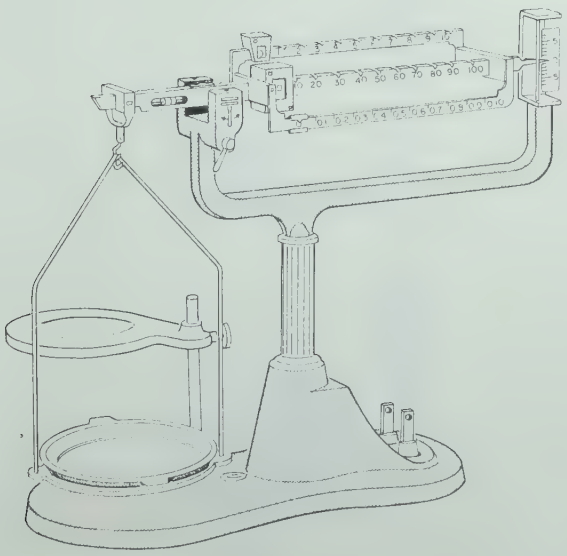


Fig. 1-4

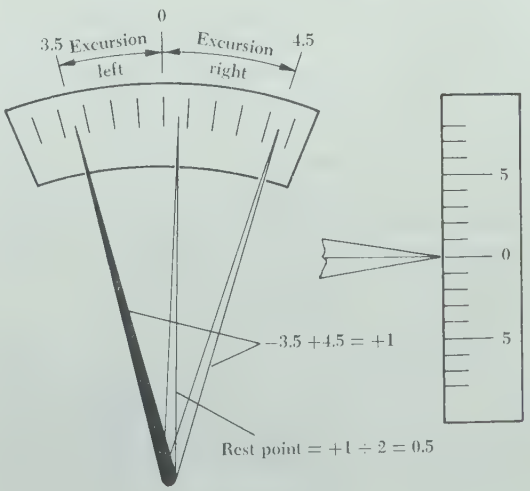


Fig. 1-5

is reached. Such rough weighings are not usually recorded.

4. *Handling solids.* Solids are usually kept in wide-mouthed bottles. A porcelain or plastic spoon may be used to dip out the solid. *Never use more of a chemical than directed.*

In order to transfer a solid to a test tube, first place it on a piece of glazed paper about 10 cm square. Roll the paper into a cylinder and slide it into the test tube as it lies flat on the table. When you lift the tube to a vertical position, and tap the paper gently, the solid will slide down into the test tube. *Never try to pour a solid from a bottle into a test tube. As a precaution against contamination, never pour unused chemicals back into the reagent bottles without specific permission from the instructor.*

5. *Measuring liquids.* For approximate measurements, a graduated cylinder, as shown in Fig. 1-6, is generally used. These cylinders are usually graduated in milliliters (ml). Such a graduate may read from 0 to 10 ml, 0 to 25 ml, 0 to 50 ml, or more, from bottom to top. It may also have a second row of graduations reading from top to bottom.

For more accurate measurements, either the pipet or the buret is used. See Figs. 1-7, 1-8, 1-9. The jet end of the pipet is thrust into the liquid, and the air is withdrawn from the pipet. When the liquid rises slightly above the graduated mark, a finger is placed quickly over the upper end of the pipet. As a little air is let in at the top, the liquid level falls slowly to the etched line. It is then held at that level, with the finger firmly against the top, and transferred to the vessel into which it is being measured. A bulb syringe is used with corrosive or poisonous liquids. Otherwise the mouth is commonly used to withdraw the air from the pipet. Pipets are made in many sizes and are used to deliver measured volumes of liquids.

Burets, fitted with either a stopcock, a pinch clamp, or a glass bead, are used for withdrawing any desired quantity of liquid to the capacity of the buret. Many burets are graduated in *tenths of milliliters.* In using a buret, you clamp it in position on a ring stand, fill it with liquid, and then draw off enough liquid to fill the tip below the stopcock and bring the level of the liquid down to the scale. The height at which the liquid stands is then read accurately.

Observe that the surface of such a liquid is slightly curved, concave if it wets the glass, and convex if it does not wet the glass. Such a curved surface is called a *meniscus*. If the liquid wets the glass, you read to the bottom of the *meniscus*, as shown in Fig. 1-10. Your eye must be looking along the horizontal line AC at the bottom of the curve. If you look along the line BC or DC, you will get an incorrect reading.

Fig. 1-6

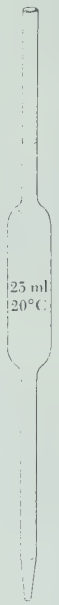


Fig. 1-7

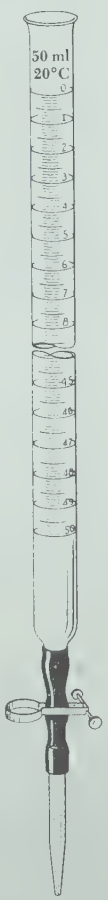


Fig. 1-8

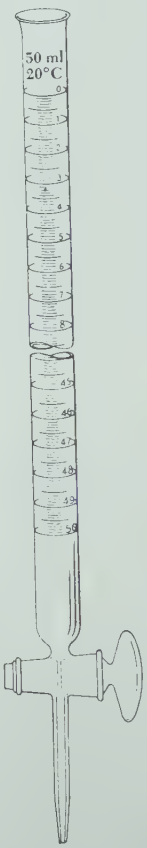


Fig. 1-9

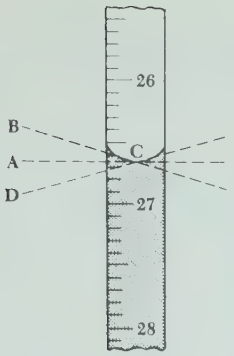


Fig. 1-10

After you have taken your first buret reading, as directed, open the stopcock and draw off as many milliliters of the liquid as you wish. The exact amount drawn off is equal to the difference between your first and final buret readings. Practice measuring liquids by measuring 10 ml of water, first using a graduate, then a pipet, and finally a buret.

6. *Assemble a wash bottle.* Use 6 mm glass tubing to form the mouthpiece and delivery tube. See Fig. 1-11 for assembly details. Observe that the angles to be formed by the tubes are mutually supplementary. You may arrange a guide to use in forming these angles by drawing a *tilted T* on your asbestos square. Use a protractor to form the 60° -120° angles accurately. Form the bends in the tubing before cutting to length and fire polishing. Insert the delivery tube through the 2-hole No. 5 stopper before forming the slight bend shown below the base of the neck of the flask. Why? Why should the mouthpiece tube terminate in the neck of the flask just beyond the stopper? Connect one of your jet tips to the delivery tube by means of a small section of rubber tubing. Fill the flask to the base of the neck with distilled water and assemble your wash bottle. See Fig. 1-12 for the proper method of holding and using the wash bottle. It has many uses in the laboratory. Your instructor will give you instructions as to its proper storage.

7. *Filtration.* Sometimes liquids contain particles of insoluble solids, either present as impurities or as precipitates formed by the interaction of the chemicals used in the experiment. If they are denser than water, they soon sink to the bottom. Most of the clear, supernatant (swimming above) liquid may be poured off without disturbing the precipitate. Such a method of separation is known as *decantation.*

Fine particles, or particles which settle slowly, are often separated from a liquid by *filtration.* Support a funnel on a small ring on the ring stand as shown in Fig. 1-12, with the stem of the funnel just touching the inside wall of the beaker. Use a beaker to collect the *filtrate.*

Fold a circular piece of filter paper along its diameter, and then fold it again to form a quadrant. See Fig. 1-13. Separate the folds of the filter, with three thicknesses

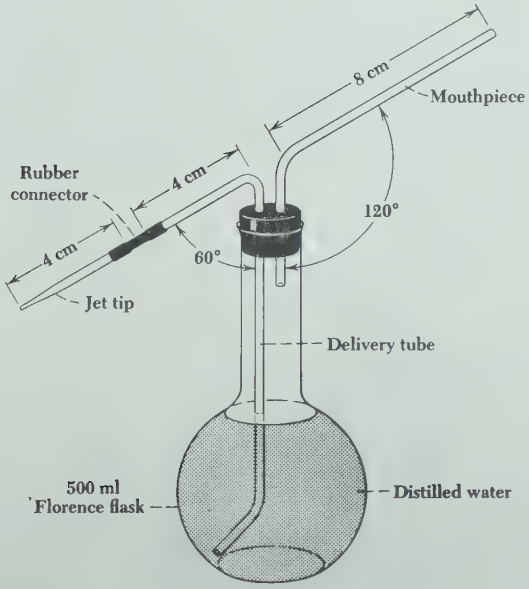


Fig. 1-11

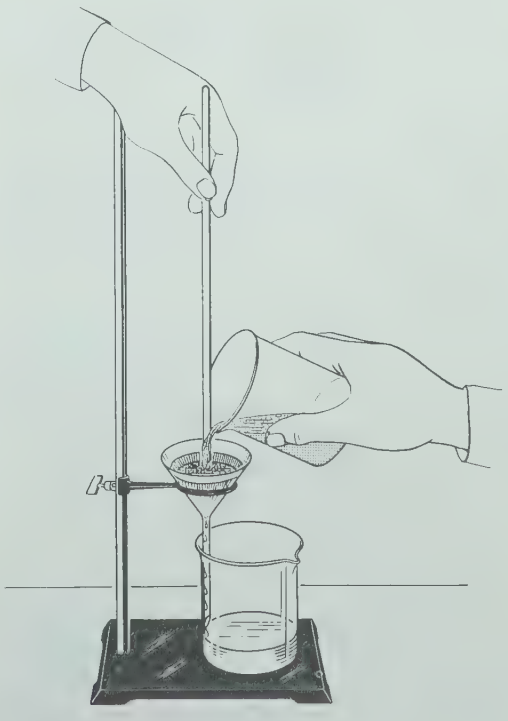


Fig. 1-12

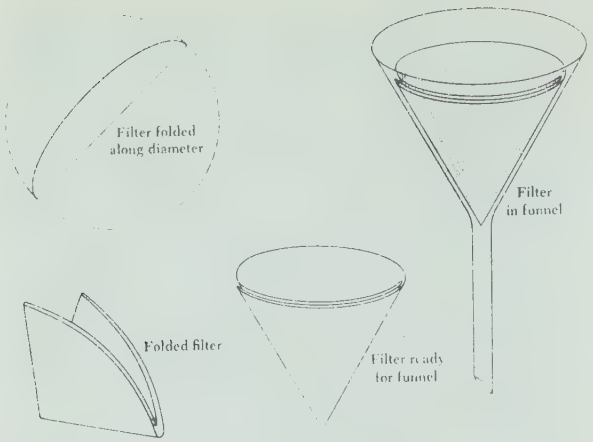


Fig. 1-13

on one side and one on the other, then place in the funnel. The funnel should be wet before the paper is added. Use your wash bottle. Then wet the filter paper with a little water and press the edges firmly against the sides of the funnel so no air can get between the funnel and the filter paper while the liquid is being filtered. **EXCEPTION:** *A filter should not be wet with water when the liquid to be filtered is not miscible with water. Why?*

Dissolve 2 or 3 g of salt in a beaker containing 50 ml of water, and stir into the solution an equal bulk of fine sand. Then filter out the sand by pouring the mixture into the filter, observing the following suggestions.

a. The filter paper should not extend above the edge of the funnel. It is better to use a filter disc which leaves about 1 cm of the funnel exposed.

b. Do not fill the filter. It must never overflow.

c. Try to establish a water column in the stem of the funnel, thus excluding air bubbles, and then add the liquid just fast enough to keep the level about 1 cm from the top of the filter.

d. When a liquid is poured from a beaker or other container, it may adhere to the glass and run down the outside wall. This may be avoided by holding a stirring rod against the tip of the beaker, as shown in Fig. F-8. The liquid will run down the rod and drop off into the funnel without running down the side of the beaker.

The sand suspended from the liquid is retained on the filter paper. What property of the sand enables it to be separated from the liquid by filtration? What does the filtrate contain? The salt may be recovered from the filtrate by pouring it into an evaporating dish, and evaporating it *nearly* to dryness. Remove the flame as soon as the liquid begins to spatter. What property of the salt prevents it from being separated from the water by filtration?

8. *Assembling apparatus.* Gases are usually generated in flasks or bottles and collected by water displacement, or if they are soluble, by air displacement. Vapors may be collected by condensation. A simple distilling apparatus to illustrate this method is shown in Fig. 1-14. The funnel tube and glass bend should be assembled through the 2-hole stopper (No. 4) before the stopper is placed in the neck of the 250-ml Florence flask. Why? Wet the glass surface and work the tubing through the stopper slowly with a twisting motion. Have both hands covered with a towel. See Fig. F-1 for safety precautions. The elbow tube extends just through the stopper and the funnel tube extends to within about 1 cm of the bottom of the flask. Clamp an iron ring on the ring stand at the proper height to allow the bottom of the flask to be just above the tip of the inner cone of the burner flame. Place a wire gauze on the ring and clamp the flask in place resting on the gauze. **CAUTION:** *Avoid clamping the neck of the flask more tightly than necessary to provide proper support. Why? Be sure the flask is Pyrex. Why?* How can you identify such glass? Next mount the funnel-tube-and-elbow assembly and attach a suitable length of rubber delivery hose. *Note that the ring, clamp, and flask are directly above the base of the ring stand.* Reason? Cut and fire polish a straight piece of 6-mm glass tubing slightly longer than the test tube to be used to collect the *distillate*. Place the test tube in a 400-ml beaker of cold water, attach the cooled glass tube to the delivery hose, and extend it to the bottom of the test tube.

Add a single crystal of potassium permanganate to 100 ml of water. Stir until dissolved; then pour into the flask by way of the funnel tube. Apply heat and bring the solution to boiling. As boiling begins, turn

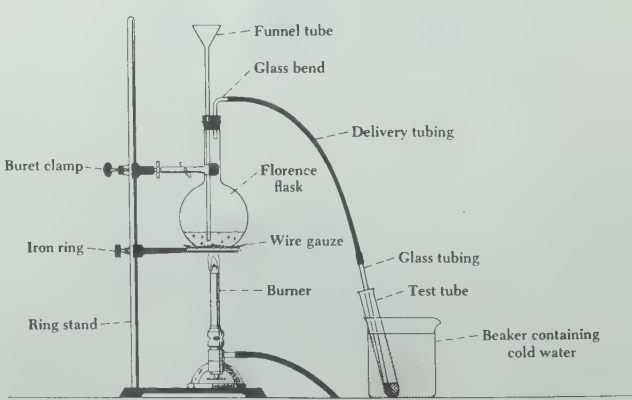


Fig. 1-14

down the flame to just sustain the boiling action. What collects in the test tube? In what way does it appear to be different from the solution in the flask? How do you explain this difference?

After collecting several milliliters of distillate, shut off the burner and allow the apparatus to cool sufficiently to be disassembled. When it is cool, disassemble the apparatus, dry and put away. Always completely disassemble glass tubing in contact with rubber stoppers and hose immediately after use. Why? Have the glass surface thoroughly wet. Follow the same precautions as in assembly. Equipment stored in your locker should be clean, dry, and arranged in an orderly fashion. Reason?

9. *Measuring temperature and heat.* A thermometer (Fig. 1-15) is used to measure temperature and temperature changes. Examine your thermometer and the temperature range for the Celsius temperature scale. Compare the Celsius temperatures with those on the Fahrenheit scale which appear on the thermometer in the laboratory.

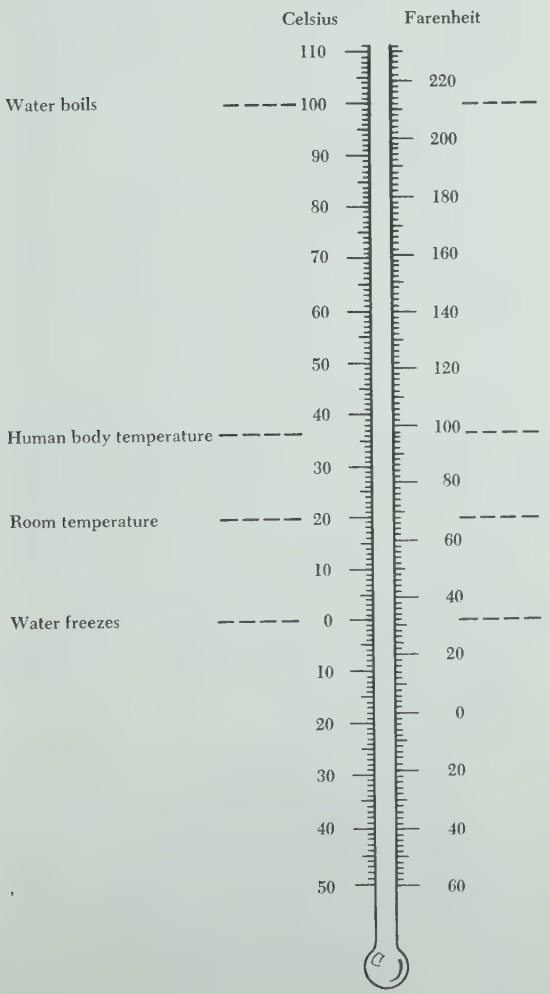


Fig. 1-15

A calorimeter is an apparatus used in measurements involving heat, and heat transfer. For approximate measurements, a simple calorimeter, as shown in Fig. 1-16, may be used. The small quantities of heat which may be transferred to or from the calorimeter by its contents may be disregarded because of the insulating characteristics of the plastic material of which it is composed. Adjust the position of the thermometer in the calorimeter lid so that the bottom of the thermometer is approximately 1.0 cm above the bottom of the inside cup.

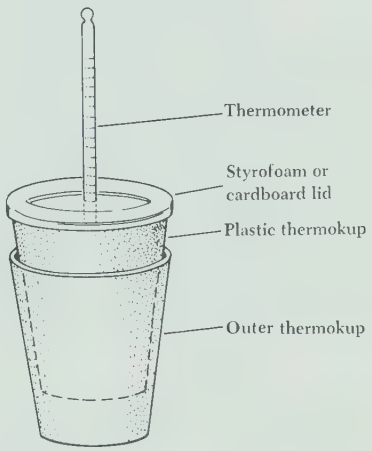


Fig. 1-16

Pour 50 ml of tapwater, measured in a graduated cylinder, into your calorimeter and record its temperature in the Data Table (p. 104). Pour 50 ml of water into a 250 ml beaker. Warm the water to 60° C using the apparatus set up as in Fig. F-6 but omit the watch glass. Record the temperature of the warm water. Immediately transfer the warm water into the calorimeter cup. Replace the lid, insert the thermometer, and stir the contents gently until a fixed temperature is reached. Record this final temperature of the mixture of hot and cold water in the Data Table. The quantities of heat, Q, in calories, gained by the cold water and lost by the hot water may each be calculated from the equation:

$$Q = \text{temperature change } (C^\circ) \times \text{mass (g)} \times 1.0 \text{ cal/g } C^\circ.$$

Using this equation, calculate Q for the heat gained by the 50 g of cold water and Q for the heat lost by the hot water. Record these quantities in the Data Table. How do these two quantities of heat compare?

DATA TABLE

Mass of cold water	g
Temperature of cold water	°C
Mass of hot water	g
Temperature of hot water	°C
Fixed temperature of mixture	°C
Temperature change of cold water (Fixed temperature — initial temperature)	C°
Temperature change of hot water (Initial temperature — fixed temperature)	C°
Heat gained by cold water	cal
Heat lost by hot water	cal

10. *Precision.* Precision is the agreement between the numerical values of two or more measurements that have been made in the same way. Precision refers to the reproducibiltiy of measurement data or the degree of detail — that is, the ± uncertainty — involved. The reproducibility of a measurement would not ordinarily be expected to exceed the tolerance of the instrument used. Carefully inspect the instruments listed below. Then record their typical tolerances as ± uncertainities.

DATA TABLE

Instrument	Uncertainty
Platform balance	g
Centigram balance	g
50 ml buret	ml
10 ml graduated cylinder	ml
50 ml graduated cylinder	ml
15 cm ruler (grad. in mm)	cm

QUESTIONS: *Answer in complete sentences.*

1. What may cause the laboratory burner to strike back? Too much air, too little gas, or dirt clogging the valve.

2. State two reasons why the luminous flame is less desirable than the nonluminous flame as a source of heat in the laboratory.

(a) The luminous flame deposits soot.

(b) It is not as hot as the nonluminous flame.

3. How is a piece of glass tubing cut into two pieces? The tubing is scratched with a file at the desired point, the thumbs are placed together on either side of the scratch, and both ends of the tube are pulled inward with the fingers.

4. How is a glass bend fire polished? The cut end is held in the hottest part of the flame until softened, causing the sharp edge to be rounded.

5. When using a platform balance of the type shown in Fig. 1-3, what determines the proper platform to be used to support the material the mass of which is to be measured? The position of the index on the slider scale determines which platform receives the material whose mass is to be measured.

6. List three instruments used in the laboratory for measuring small quantities of liquids. Liquids are measured in graduated cylinders, pipets, and burets.

7. What is the rule about size of filter paper for use with a given funnel? The funnel should extend about one cm above the edge of the filter cone.

8. How can a liquid be transferred from a beaker to a funnel without spattering, and without running down the outside wall of the beaker? The stirring rod may be held against the lip of the beaker, and the liquid will cling to the rod.

9. Why must the end of the funnel tube dip down into the liquid boiling in a flask? The tip of the funnel tube must extend into the liquid to prevent the vapor from escaping through the funnel tube rather than through the delivery tube.

10. What is the advantage of determining the *rest point* of a sensitive balance and using it in carrying out a precise weighing procedure? Determining the rest point saves time in weighing. It is not necessary to adjust the balance each time it is used so as to cause the center of the range over which the pointer swings to coincide with the center of the fixed scale.

11. Compare the quantities of heat loss and heat gained? Is one greater? If so, why? Theoretically the heat lost equals the heat gained, however, some heat may be lost to the atmosphere.

NAME . . . . . PERIOD . . . . . DATE . . . . . .
INSTRUCTOR'S COMMENTS . . . . . .

Experiment 2 Measurements and Accuracy

PURPOSE: To measure various physical properties of matter and to determine the accuracy of such measurements.

APPARATUS: Balance, platform; beaker, 100 ml; graduated cylinder, 50 ml; ruler, 15 cm, plastic; thermometer.

MATERIAL: Metal shot, aluminum, copper, lead.

INTRODUCTION: In this experiment the volume of the graduated cylinder to the 50-ml graduation will be calculated from measurements of its internal diameter and height to the graduation. The mass of a measured volume of water will be calculated from its density ($m = V \times D$) and then determined by weighing. The density of a metal will then be calculated from measurements of its mass and volume ($D = m/V$). You will calculate the error and percentage error in each part of the experiment on the basis of accepted values.

PROCEDURE: 1. Examine the centimeter scale of the plastic ruler. What are the smallest divisions? To what fraction of a centimeter are you expected to be able to make a measurement with such a ruler?

Measure the inside diameter of the graduated cylinder using the ruler. Similarly measure the inside height of the cylinder to the 50-ml graduation. Record these observations in the Data Table. Calculate the volume of the cylinder to the 50-ml graduation ($V = 3.14\ r^2 \times h$). Assume the capacity in ml of the cylinder (to the top graduation) to be equal to that numerical value in cm^3. Using this value in cm^3 as the *accepted value*, calculate the error and percentage error.

DATA TABLE

Inside diameter of cylinder	cm
Inside height of cylinder	cm
Volume of cylinder (computed) . .	cm^3
Volume of cylinder (accepted) . . .	cm^3
Error	cm^3
Percentage error	% %

2. Examine the 0-10 gram scale of the platform balance. What are the smallest divisions? To what fraction of a gram may you make measurements with a platform balance? Is this compatible with its uncertainty?

Similarly examine the graduation on the graduated cylinder and determine the smallest fraction of a millimeter to which you could make a measurement? Is this compatible with the uncertainty of a 50-ml graduated cylinder?

Using the platform balance, determine the mass of the dry cylinder. Record the mass in the Data Table. Half fill the beaker with water and determine its temperature to the nearest degree. Look up the density of water for this temperature and record both the temperature and water density. Fill your graduated cylinder to some value between 40 and 50 ml, accurately read and record the volume. Determine the mass of this volume of water plus that of the cylinder by weighing. Then record. Calculate the mass of the water as determined by use of the balance. Calculate the mass of the water from its measured volume and its density ($m = D \times V$). Record these values in the Data Table. Using the mass determined by the use of the balance as the *accepted value*, calculate the error and percentage error.

DATA TABLE

Mass of cylinder and water	g
Mass of empty cylinder	g
Mass of water	g
Measured volume of water	ml(cm^3)
Temperature of water.	°C
Density of water.	g/cm^3
Mass of water ($m = D \times V$)	g
Error	g
Percentage error	%

3. Pour out some of the water in the cylinder until it contains between 25 and 30 ml. Measure and record this volume in the Data Table. Determine the mass of the cylinder and the water and record. Add a sufficient quantity of the assigned metal shot (aluminum, copper, or lead) to the cylinder containing the water to increase the volume by at least 10 ml. Determine the volume and then the mass of the shot, water, and

cylinder and record your measurements. Calculate the volume of the metal shot, its mass, and its density. Record the results of these calculations. Look up the specific gravity of the metal in a Handbook or your textbook Appendix. The density of liquids and solids in the metric system is numerically equal to the specific gravity. Record this *accepted value*. Calculate the error and percentage error.

DATA TABLE

Volume of metal shot and water	ml
Volume of water	ml
Volume of metal shot (computed)	ml
Mass of metal shot + water + cylinder	g
Mass of water + cylinder	g
Mass of metal shot	g
Density of metal (computed: 1 ml = 1 cm^3)	g/cm^3
Density of metal (accepted)	g/cm^3
Error	g/cm^3
Percentage error	%

4. Precision is the agreement between the numerical values of two or more *measurements* that have been made in the same way. It is expressed in terms of *deviation*. An *absolute deviation* D_a is the difference between an observed value O and the arithmetic mean (average) M for set of identical measurements.

$$D_a = O - M$$

In the Data Table record at least five results compiled by you and your classmates for the density of the same metal. Calculate the average for these results, and enter it in the Data Table. Compute the absolute deviation D_a for each measurement and enter these quantities in the Data Table. Do not enter the sign of the deviation. Calculate the average value of the deviations and enter this quantity at the bottom of the Deviation column in the Data Table. This quantity with a ± sign before it is the *uncertainty* in the measurements data. Now in the appropriate place in the Data Table express the result of this set of measurements as M ± average deviation.

DATA TABLE

Experiment	Measurement (Density: g/cm^3)	Deviation (D_a)
1		
2		
3		
4		
5		

Average: M =

Uncertainty = ± g/cm^3

Expression for set of experimental results = M ± uncertainty = ± g/cm^3

QUESTIONS: *Answer in complete sentences.*

1. What value of a measurement must be available if the accuracy of a measurement is to be determined? The accepted value of a measurement must be available.

2. What type of error must be calculated before the percentage error may be determined? The absolute error (experimental error) must be calculated.

3. What are the possible sources of experimental errors in this experiment? Sources of experimental error include inaccurate observation and imperfections in apparatus design which makes the use of measuring devices difficult (as in Part 1.)

4. Why is it impossible to determine uncertainties in terms of precision in this experiment as it was performed? Only one set of measurements, made in the same way, was made.

NAME. . . . . PERIOD . . . . . DATE . . . . . .
INSTRUCTOR'S COMMENTS . . . . . .

Experiment 3

General Classes of Matter

PURPOSE: To recognize some differences in properties of two elements and between their mixture and a compound formed from these elements.

APPARATUS: Balance, platform; bar magnet; beaker, 250 ml; burner and tubing; clamp, test tube; funnel; graduated cylinder; magnifying lens; ring, iron; ring stand; stoppers, cork, to fit test tubes; test tubes; test tube, Pyrex; watch glass.

MATERIAL: Iron metal filings, 60–80 mesh; sulfur, powdered; filter paper; hydrochloric acid, dilute (1:4); carbon disulfide.

INTRODUCTION: In this experiment we shall use two common elements, iron and sulfur. We shall form a mixture of these elements and compare its properties with those of the elements themselves. Then we shall cause the elements to react to form a compound, and again compare the properties with those of the constituent elements.

PROCEDURE: 1. Spread approximately 5 g of iron filings on a small square of glazed paper. Try the effect of a magnet by passing the magnet back and forth under the paper. Result? Repeat this operation, using approximately 3 g of powdered sulfur. Result? Save these two quantities for use in Part 4.

Observations: Record what you observe briefly but accurately.

Iron filings are attracted by the magnet. The powdered sulfur is not attracted by the magnet.

2. Measure out 10 ml of carbon disulfide and idvide it equally into two test tubes. To one test tube, add 0.5 g of powdered sulfur; and to the other, add 0.5 g of iron filings. Stopper both test tubes and shake them gently to see if carbon disulfide will dissolve either of these substances. Results? **CAUTION:** *Keep carbon disulfide away from open flames as it is combustible! Avoid inhaling its vapors.*

Observations: Sulfur dissolves in carbon disulfide, iron does not.

3. Add 5 ml of dilute hydrochloric acid to each of two test tubes. To one, add 0.5 g of powdered sulfur; to the other 0.5 g of iron filings. In which tube(s) do you observe evidence of a chemical reaction? Describe. Test for odor of any gaseous product. **CAUTION:** *See Fig. F-5 for the proper technique of investigating odors.*

Observations: A gas (H_2) is liberated as the iron reacts with the hydrochloric acid.

4. Mix thoroughly on a square of glazed paper the sulfur and iron filings of Part 1, using a spatula or stirring rod. Try the effect of a magnet on the mixture, again moving the magnet back and forth under the paper. Result? Inspect the mixture, using a magnifying lens. Describe. Remix and transfer half of the mixture to a test tube. Add 10 ml of carbon disulfide; stopper and shake vigorously. Filter the contents (dry filter paper), catching some of the filtrate on a watch glass. Recall the solubility test made in Part 2. What would you expect the filtrate to contain? Place the watch glass in the hood or on the ledge of an open window and allow the liquid to evaporate. Examine the residue with the magnifying lens. Were your expectations confirmed? Describe the residue. What does the residue remaining on the filter paper appear to be? How could you confirm this based on the characteristics of the elements you have observed?

Observations: The iron filings are attracted to the magnet. Separate individual particles of iron and sulfur are detected by the magnifying lens. The filtrate should contain sulfur. Evaporation of filtrate confirms the expectation and results in small crystals of solid sulfur. These will not be attracted by the magnet but will redissolve in carbon disulfide.

5. To the other half of the mixture, Procedure 4, add 5 ml of hydrochloric acid in a test tube. Result? What do the results of Procedures 3 and 4 indicate about the effect of mixing on the properties of the elements involved?

Observations: A gas (H_2) is liberated by the reaction between the iron and the hydrochloric acid. This result indicates that the properties of the elements are not changed by mixing them.

6. Mix 7 g of iron filings and 4 g of powdered sulfur as before, transfer to a dry Pyrex test tube, and heat strongly until a definite red glow is observed within the contents of the tube. Remove the tube from the flame. Does the reaction stop? When all evidence of a reaction ceases, break the test tube by plunging the hot end into a beaker of cold water. Recover the solid product and examine it. Try the effect of a magnet. Result? Examine it closely with the magnifying lens. Result? Try the effect of 3 ml of carbon disulfide on it. Result?

Observations: The reaction does not stop, the red glow continues up through the test tube contents. The magnet does not attract the product. Carbon disulfide does not dissolve the product.

7. To 5 ml of hydrochloric acid in a test tube add a small piece of the product obtained in Part 6. Describe any evidence of chemical reaction. Note any difference in odor of an observed gaseous product and compare with the results of the odor test in Part 3.

Observations: Gas production occurs only with the iron filings and the product. The gas resulting from the acid on the product has the rotten egg odor of hydrogen sulfide.

QUESTIONS: *Answer in complete sentences.*

1. What are three ways of distinguishing between iron and sulfur?

a. A magnet attracts iron but not sulfur.

b. Sulfur dissolves in carbon disulfide; iron does not.

c. Sulfur is yellow; iron is dark gray in appearance.

2. How do you know that the iron has not united with the sulfur when these two elements are merely mixed together? The magnet attracts the iron and separates it from the sulfur. Carbon disulfide dissolves the sulfur in the mixture.

3. In what proportions can a mixture of these two elements be made? The mixture of iron and sulfur can be made in any proportion of the elements.

4. What evidence did you observe that the iron and sulfur reacted when the mixture was heated? The evolution of heat and light is evidence of a chemical reaction.

5. What is necessary to cause the iron and sulfur to react? Heat is necessary to cause the reaction. It must be supplied externally to start the reaction.

6. Why were you directed to use definite quantities of iron and sulfur for preparing the compound of these two elements? The elements unite in definite proportion.

7. How did the hydrochloric acid test show that you had produced a compound with different properties from thsoe of either iron or sulfur? The "rotten egg" odor of the gas produced in one tube indicated that a new compound was reacting with the hydrochloric acid.

NAME . . . . . PERIOD . . . . . DATE . . . . . .
INSTRUCTOR'S COMMENTS . . . . . .

Experiment 4

Physical and Chemical Changes

PURPOSE: To study the differences between physical and chemical changes in matter.

APPARATUS: Asbestos square; beakers, 100 ml, 150 ml; burner and tubing; forceps; funnel; ring, iron; ring stand; test tubes; thermometer; wash bottle; watch glass; wire gauze.

MATERIAL: Alka-seltzer tablets; baking powder; copper foil; magnesium ribbon; platinum wire test rod; sandpaper, fine grit; zinc, mossy; filter paper; hydrochloric acid, dilute (1:4); sulfuric acid, dilute (1:6); solution of silver nitrate (0.2 *M*); wooden splints.

SUGGESTION: Since fresh mossy zinc may react very slowly, pieces previously used and collected may be used. Provide receptacle(s) for any residual zinc pieces.

INTRODUCTION: Matter undergoes many changes. In some cases only the temperature, physical state, size of particle, or color is changed. Ice melts and water evaporates. *Such changes are physical.*

In other cases different substances with new characteristic properties are formed. Wood burns and metals tarnish. *Such changes are chemical.* Heat, light, electricity, and solution are often instrumental in *starting* chemical changes. In many cases, too, they are produced as the immediate result of such changes.

PROCEDURE: 1. Examine the platinum wire test rod. Observe the color and luster of the metal. Hold the wire in the flame of your burner for about two minutes. Recall Part 1 of Experiment 1 in which you determined the hottest part of the flame. Does the appearance of the platinum wire support your previous conslusion? Describe the appearance of the wire while held in the hottest part of the flame. Allow the wire to cool and re-examine it. Conclusion?

Observations: Platinum wire has a silvery luster. The wire glows brightly in the hottest part of the flame. Upon cooling, its appearance is the same as before heating. Therefore it did not undergo a chemical change.

2. Sandpaper a piece of magnesium ribbon about 3 cm in length to remove the tarnish. Note the color, luster, and flexibility of the metal. Perform the next operation over the asbestos square. Holding one end with the forceps, ignite the other end in the burner flame. CAUTION: *Do not look directly at the magnesium while it is burning.* Compare the ash, which may be collected on the asbestos square, with the original metal.

Observations: Student answers.

3. Similarly clean a piece of copper foil and heat it in the outer cone of the burner flame for 1–2 minutes. Avoid melting. Let it cool and re-examine it. See if you can scrape off some of the black scale from the surface of the copper. Compare the properties of this scale with those of metallic copper. Heat the foil a second time. Result? Explain.

Observations: The black scale is readily removed and evidently is not copper. Another layer of the CuO forms when the cleaned foil is reheated. The copper undergoes a chemical change since the product has different properties.

4. Place a thermometer into the liquid in a test tube containing 4 or 5 ml of dilute sulfuric acid. Observe carefully for any signs of chemical action. Result? Remove the platinum wire, flushing it with water before laying aside. Add a small piece of zinc to the acid. Observe carefully for any signs of chemical action. Result? Let the action continue for about 5 minutes. Remove the thermometer and warm the tube gently, CAUTION.

When the reaction is proceeding vigorously, bring the flame of a burning splint to the mouth of the test tube. Result? The dilute sulfuric acid is a water solution of hydrogen sulfate. What gas do you think is being evolved during the reaction? Account for the appearance of a black suspension in the liquid. Filter to remove suspended matter, if present. Do you believe a second product of the reaction to be in solution

in the filtrate? Using an appropriate technique, recover this product from a portion of the filtrate. Compare the product with the original acid and with the zinc. Suggest the probable name of the crystalline substance. Try writing a word equation to express your idea of the chemical reaction that has taken place.

Observations: Student answer.

5. To 3 or 4 ml of silver nitrate solution, add several drops of dilute hydrochloric acid. Result? Can you recognize a *precipitate*? Describe it. Hydrochloric acid is a water solution of hydrogen chloride. Consult the solubility table in your textbook and suggest the probable name of the insoluble product (the precipitate). Try writing a word equation to express your idea of the chemical reaction that has taken place. Was there any evidence of a gaseous product of this reaction? Transfer the entire contents of the test tube to a filter setup, flushing with small additions of water from the wash bottle, if necessary, to remove all of the precipitate. Discard the filtrate, unfold the filter paper and expose the precipitate to direct sunlight for several minutes. Result? Do you think this change is physical or chemical? Did you observe any evidence of an appreciable energy change during the reaction?

Observations: Student answer.

6. To one-half an Alka-seltzer tablet or to 2 grams of baking powder in a 100 ml beaker add 5 ml of water. Result? After the reaction proceeds for 15 seconds, thrust a burning splint into the upper portion of the beaker. Result? Both Alka-seltzer and baking powder contain a solid substance, which acts as an acid, and sodium hydrogen carbonate. What gas do you think is being evolved during the reaction? Why did the chemicals which did not react in the dry state begin to react?

Observations: A gas carbon dioxide is produced. The burning splint is extinguished in the carbon dioxide.

QUESTIONS: *Answer in complete sentences.*

1. What kind of change occurs when platinum is heated? Physical

2. Is a new substance formed as magnesium burns? Yes. Justify your answer. The product is a white powder unlike the magnesium metal.

3. How does the scale which forms when copper is heated differ from the copper? The scale is black and brittle; copper is lustrous and flexible.

4. What do you think would be the ultimate result of successive heatings and scrapings of the copper? The copper would finally be converted entirely to the black scale.

5. Does platinum interact with sulfuric acid? No.

Does zinc interact with sulfuric acid? Yes.

Do the crystals formed in Part 4 resemble either zinc or sulfuric acid? No, a new substance has been formed.

6. Does the precipitate in Part 5 seem to be a new substance? Yes. Is this a chemical or physical change? It is a chemical change.

7. The precipitate in Part 5 is silver chloride. What effect does sunlight have on the precipitate? The precipitate darkens.

Do you think such a change may have some relation to photography? Yes, light affects a silver compound on the film.

8. Summarize the results of this experiment in a definite, concise conclusion. Physical changes do not produce a new substance. Chemical changes always produce one or more new substances.

NAME . . . . . PERIOD . . . . . DATE . . . . . .
INSTRUCTOR'S COMMENTS . . . . . .

Experiment 5 Oxygen: Catalysis

PURPOSE: To show that oxygen may be prepared from a number of chemical compounds and to show how catalysts play a part in chemical reactions.

APPARATUS: Balance, platform; clamp, buret; clamp, test tube; funnel tube; glass bend; 2 glass plates; graduated cylinder; ring stand; rubber stoppers, solid No. 3, 2-hole No. 4; test tubes, Pyrex (6), 200 mm, Pyrex (1); trough; tubing, delivery.

MATERIAL: Barium peroxide; calcium carbonate, powdered; charcoal, activated, pellets; lead(II)(IV) oxide; lead(IV) oxide; manganese dioxide C.P.; mercury(II) oxide; potassium permanganate; sodium peroxide; litmus papers, red and blue; wooden splints; yeast, dried; solution of hydrogen peroxide (6%). Optional: copper(II) oxide, powdered; iron(III) oxide; potassium chlorate, C.P., potassium dichromate.

INTRODUCTION: Oxygen may be prepared conveniently from other substances beside potassium chlorate without heating. These include sodium peroxide and hydrogen peroxide. A variety of substances will be added to hydrogen peroxide to determine their possible use as catalysts in bringing about its decomposition.

SUGGESTIONS: The instructor may wish to demonstrate the role of a catalyst in the decomposition of potassium chlorate. A 3–5 gram sample of potassium chlorate is heated until decomposition starts and the presence of oxygen is demonstrated with a glowing splint. The sample is permitted to cool until the evolution of oxygen apparently stops. The addition of 0.5 g of powdered manganese dioxide causes the reaction to occur at this lower temperature and the glowing splint test should be used again. The same procedure may be used to demonstrate the possible use of other catalysts: copper (II) oxide; iron(III) oxide; and potassium dichromate.

PROCEDURE: 1. Place 3 g of manganese dioxide in the large test tube using the technique of Experiment 1, Part 4. Clamp the test tube to the ring stand. Attach the rubber delivery tube to the two-hole stopper, funnel tube, glass bend assembly, and insert this assembly into the test tube, Fig. 5-1. Fill a test tube with water, cover the end with a glass plate, and place it mouth downward in the trough. Add 5 ml of hydrogen peroxide solution through the funnel tube. Permitethe reaction to proceed for half a minute. Then collect a test tube of oxygen. Stopper the test tube while its mouth is still under water. Place the stoppered test

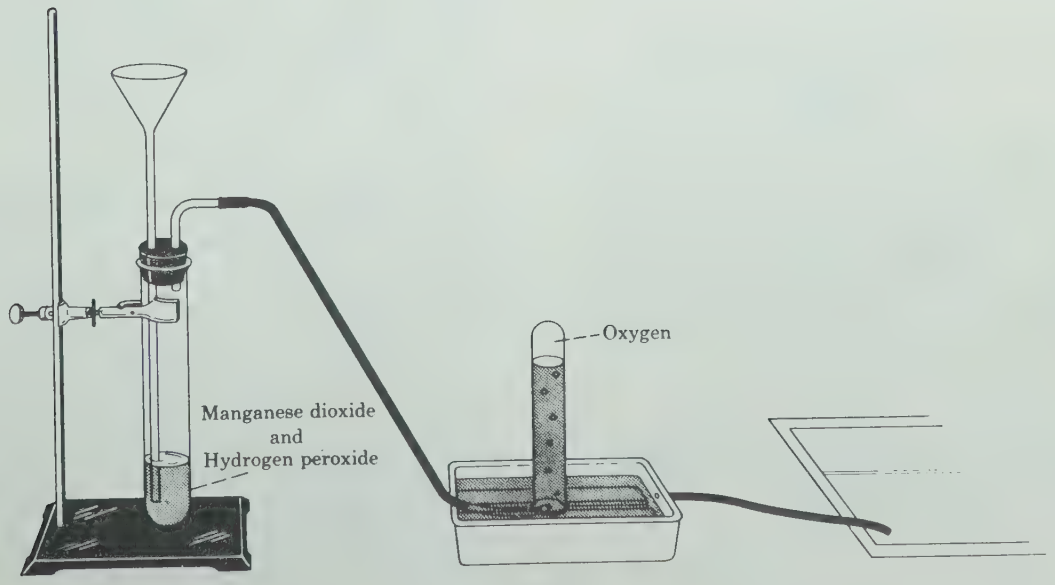


Fig. 5-1

DATA TABLE

Substance added	Evidence of evolution	Result of glowing splint test
Activated charcoal (C)	yes	positive
Potassium permanganate ($KMnO_4$)	yes	positive
Yeast	yes	positive
Calcium carbonate ($CaCO_3$)	no	negative
Lead(II)(IV) oxide (Pb_3O_4)	yes	positive

tube in the test tube rack. Why was the collection of oxygen not started immediately upon the addition of hydrogen peroxide? Light a splint, blow out the flame, and insert the glowing splint in the oxygen. What result do you observe?

Observations: Student answer.

2. Into each of five test tubes in the test tube rack pour 3 ml of the hydrogen peroxide solution. To the first test tube add 4 pellets of activated charcoal. Report any evidence of gas evolution. If a gas is produced, test for oxygen by inserting a glowing splint into the space above the hydrogen peroxide. Repeat the above procedure using, in successive test tubes, 3 small crystals of potassium permanganate, 0.5 g of dried yeast, 0.5 g of powdered calcium carbonate, and 0.5 g of lead(II)(IV) oxide. Record your observations in the Data Table.

Decant the hydrogen peroxide solution from the test tube containing the activated charcoal pellets. Add another 3 ml of hydrogen peroxide solution. Result?

Oxygen is produced again.

3. Place 1 g of sodium peroxide in a test tube. Add 5 ml of water. Result? Insert a glowing splint into the upper part of the test tube. Result? Dip a stirring rod into the test tube contents and touch the moistened end of the rod to a piece of blue litmus paper on a clean glass plate. Result? Repeat the procedure using a piece of red litmus paper. Result? (Basic solutions of metallic hydroxides turn red litmus to blue, while acidic solutions turn blue litmus red.)

Observations: A gas, O_2 is produced and the glowing splint bursts into flame. The resultant solution changes red litmus to blue, the blue litmus stays blue.

Repeat the above procedure using, in successive test tubes, 1 g each of mercury(II) oxide, lead(IV) oxide, and barium peroxide. Results?

Observations: Oxygen is only liberated from the barium peroxide.

QUESTIONS: *Answer in complete sentences.*

1. Which of the solid substances added to hydrogen peroxide caused oxygen to be produced? All, except the calcium carbonate, caused oxygen to be produced.

2. In which case or cases, if any, are you reasonably sure that the oxygen came from the hydrogen peroxide only? The oxygen definitely came from the hydrogen peroxide when activated charcoal is used as a catalyst. The other catalysts, (with the exception of yeast) are compounds containing oxygen according to their given formulas.

3. Which of the catalysts seemed to be most effective? Lead(II)(IV) oxide generally is most effective. (Students may report different ones.)

4. What characteristic of a catalyst was evident from the results of the experiment in which charcoal was used for a second trial?

Catalysts are not used up during a chemical change.

EQUATIONS

1. Hydrogen peroxide (catalytic decomposition) → water + oxygen.

2. Sodium peroxide + water → sodium hydroxide + oxygen.

NAME . . . . . PERIOD . . . . . DATE . . . . . .
INSTRUCTOR'S COMMENTS . . . . . .

Experiment 6 Energy and Entropy: Phase Change

PURPOSE: To determine the melting point of acetamide. To plot a time-temperature graph of the experimental facts and interpret them in terms of energy and entropy changes involved in a phase change.

APPARATUS: Balance, platform; beaker, 250 ml; burner and tubing; clamp, buret; clock; ring, iron; ringstand; rubber stopper, 1-hole No. 2; test tube; thermometer; wire gauze.

MATERIAL: Acetamide (practical grade).

SUGGESTION: You may wish to have half the class use another solid such as paradichlorobenzene.

INTRODUCTION: As a solid is heated its temperature rises. The continuous addition of energy finally results in a phase change, the solid melts at its melting point. Further addition of energy causes the temperature of the liquid to increase. Acetamide is a solid at room temperature. Its melting point is well below 100° C. In this experiment you will melt a sample of acetamide and record the temperature at stated time intervals as the liquid cools, solidification (crystallization) occurs, and the resultant solid undergoes subsequent cooling. The results of the experiment permit you to determine the melting point (actually the freezing point) of the substance and to interpret the changes in energy and entropy which have taken place.

PROCEDURE: 1. Place a 250-ml beaker one-half full of water on the ringstand and start warming the water. Measure out approximately 7 grams of acetamide, place in the test tube, and insert the thermometer-stopper combination into the test tube. Lower the test tube into the water bath and clamp the test tube to the ringstand (see Fig. 6-1). Heat the water until the contents of the test tube melt. To melt the acetamide, the water usually must be boiling. Remove the test tube from the heated water leaving it clamped to the ringstand, Fig. 6-2.

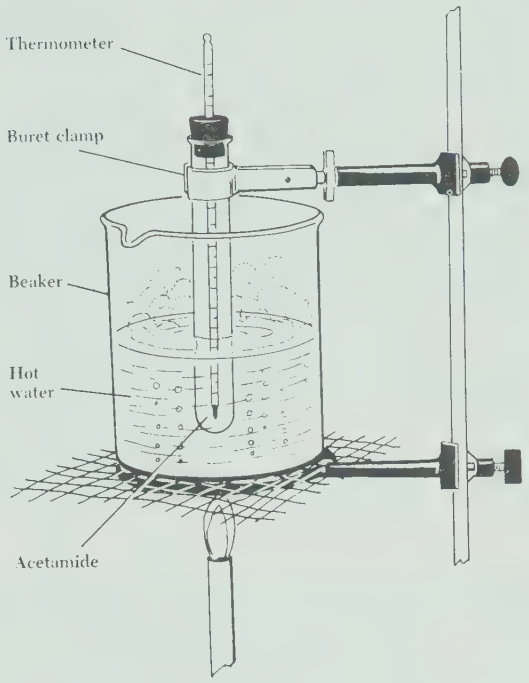


Fig. 6-1

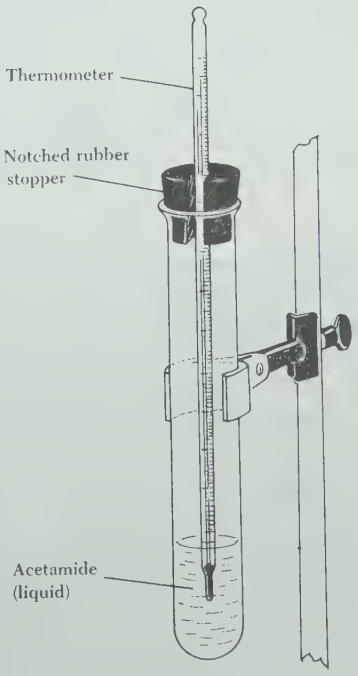


Fig. 6-2

DATA TABLE

Time (Minutes)	Temperature (°C)	Observations
0	Student answers.	
1		
2		
3		
4		
5		
6		
7		
8		
9		
10		
11		
12		
13		
14		
15		
16		
17		
18		
19		
20		
21		
22		
23		
24		
25		

2. When the temperature indicated by the thermometer is approximately 100°C, read the temperature every minute and record the readings in the Data Table. At the same time record when crystallization (freezing) starts, the places in which crystallization is taking place, and the extent of crystallization. Place these observations in the Data Table next to the appropriate times.

3. On a separate sheet of graph paper plot a graph of temperature versus time. Make time intervals the horizontal axis. Select and label the distinct portion of the graph during which crystallization (freezing) occurs.

4. If there is sufficient time, repeat the experiment using 30 second intervals.

5. Another interesting variation is to melt the acetamide, remove the thermometer, and when according to your initial data, crystallization should begin, drop several small seed crystals of acetamide into the liquid.

QUESTIONS: *Answer in complete sentences.*

1. What is the shape of the part of the graph which represents the cooling of the liquid? It slopes downward.

2. What is happening to the kinetic energy of the acetamide molecules as the liquid cools? Kinetic energy (average) decreases.

3. How does the temperature of the acetamide sample compare with the temperature of the room (its external environment) while (a) the liquid is cooling, (b) very little, if any change in temperature occurs, (c) the solid is cooling? (a) Above room temperature, (b) above room temperature, (c) may go down to room temperature.

4. In terms of the temperature differentials in question 3 are the changes taking place exothermic or endothermic? (a) exothermic, (b) exothermic, although little evidence of temperature change, (c) exothermic.

5. What happens to the average kinetic energy of the molecules of a substance as the temperature fails? The average kinetic energy decreases.

6. How would you describe the temperature change(s) that you recorded while the liquid was freezing to the solid? The temperature remained relatively constant.

7. What happens to the entropy of acetamide as it changes from a liquid to a solid? The entropy decreases.

8. In terms of the entropy-change factor and the energy-change factor, why does liquid water freeze when placed in an environment where the temperature is below 0°C? The temperature is low enough so that the entropy-change factor loses out to the energy-change factor; heat is given up by the water (lower energy) and the ordered ice crystal is formed (lower entropy).

Experiment 7

NAME . . . . . PERIOD . . . . . DATE . . . . . .

INSTRUCTOR'S COMMENTS . . . . . .

Bright-Line Spectrum of Sodium

PURPOSE: To measure the wavelength of the bright-line spectrum of sodium.

APPARATUS: Bunsen burner and tubing (asbestos collar); cardboard screen with vertical slit; card-mounted diffraction grating, 1 lens support (Welch 3604 or 3606); meter stick and half-meter stick; 4 meter stick supports (Welch 3602); 2-screen supports (Welch 3610); Optional: sodium-arc light source with mount and transformer (Welch 3720 B).

MATERIAL: Sodium chloride.

Note: The asbestos collar is made from an asbestos sheet which is saturated with sodium chloride solution, dried, and then fastened around the top of the burner with thread or fine wire.

INTRODUCTION: Examination of the bright yellow flame, characteristic of a vaporized sodium compound, through a spectroscope reveals two closely spaced yellow lines. These lines constitute the bright-line emission spectrum of sodium. They represent characteristic amounts of energy emitted when excited electrons return to lower energy levels. The existence of line spectra indicates that the energy of an electron can only be changed by its movement from one *discrete energy level* to another. These changes in energy are *quantized* (restricted to certain values). The conclusion is that the electrons in an atom have definite, distinctive energies. In this experiment, the use of the diffraction grating will make the two lines appear as one. They are very close together since their respective wavelengths are 5890 Å and 5896 Å.

The energy emitted (or absorbed) as an electron makes a jump from one particular energy level to another particular level is equal to the difference in the energies that the electron has in these two energy levels. The energy emitted or absorbed, $E = E_2 - E_1$. The energy of the photon(s) emitted or absorbed, $E = hf$. A definite frequency (f) means a definite energy, since h is Planck's constant which is identical for all types of electromagnetic radiation. The ferquency and then the energy can be calculated if the wavelength of the light is measured. The relationship which is used is c (speed of light) = f (frequency) $\times$ λ (wavelength).

PROCEDURE: 1. Arrange the apparatus as shown in Fig. 7-1. The screen support with the diffraction grating is placed on the half-meter stick at a distance of approximately 40 cm. The cardboard slit assembly is placed on the 50 cm mark of the meter stick. The meter and half-meter sticks are placed in the meter stick supports. The half-meter stick is placed perpendicular to the meter stick at the 50 cm mark.

2. Light the burner so that a continuous yellow flame is emitted by the asbestos salt-saturated collar. The burner is placed in back of the slit so that the yellow flame is clearly visible through the slit. Look through the center of the diffraction grating along the half-meter stick to locate the yellow line visible through the slit. With the eye close to the grating look to the left

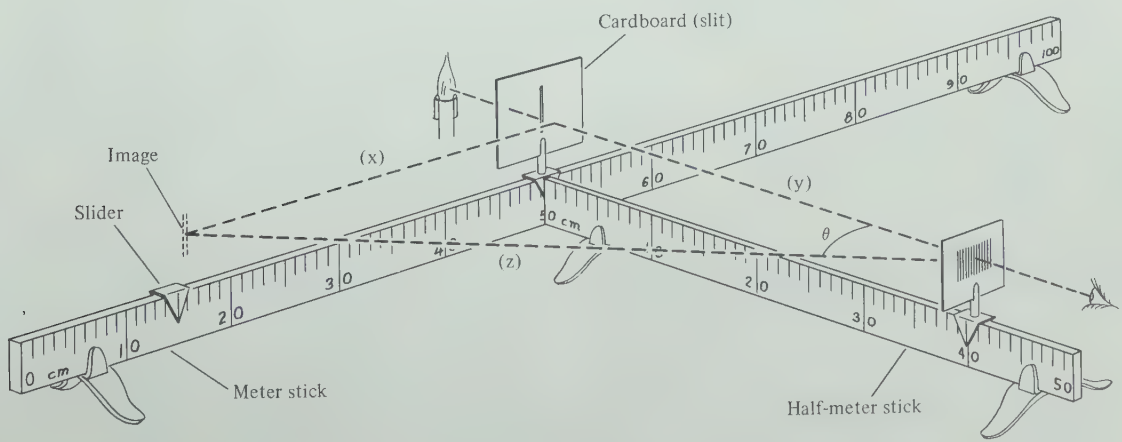


Fig. 7-1

of the yellow line to locate first order sodium spectrum (the two merged sodium spectral lines). A second student moves the slider (screen support) along the meter stick until the student looking at the spectrum indicates that the vertical line through the point of the slider coincides with the location of the spectrum.

3. The distances x and y (Fig. 7-1) are measured to the nearest millimeter and recorded in the Data Table.

4. The procedure is repeated for locating the spectrum to the right of the 50 cm mark on the meter stick. The distances x and y are measured and recorded. Time permitting, the grating distance y is changed and two more readings (left and right spectrum) are made and recorded.

CALCULATIONS: 1. The value of z is computed to the correct number of significant figures by using the Pythagorean formula: $z = \sqrt{x^2 + y^2}$. Record the values for z in the Data Table.

2. The formula to be used in calculating the wave length is $\lambda = d \sin \theta$. The value of sin θ is calculated ($\sin \theta = \frac{x}{y}$) and recorded in the Data Table.

3. The distance between two grooves of the diffraction grating (d) is calculated using the relationship, $d = \frac{2154 \text{ cm}}{13{,}400}$ since the number of grooves per inch for your grating is 13,400. Record the result in the Data Table.

4. Calculate and record the wavelength in Angstroms using the formula $\lambda = d \sin \theta$.

5. Repeat the calculations for other data that you have taken. Average these results and calculate the experimental (observed) error in Angstroms. Use 5893 Å as the accepted value. Calculate your relative (percentage) error and record in the Data Table.

DATA TABLE

First Order Spectrum	Data		Calculated Results			
	x (cm)	y (cm)	z (cm)	$\sin \theta = \frac{x}{z}$	$d = \frac{1}{N}$	$= d \sin \theta$ (Å)
1. (a) left image						
(b) right image						
2. (a) left image						
(b) right image						

Average value of λ . . . . . Student answer. . . . . Å

Experimental (observed) error . . . . . Student answer. . . . . Å

Percentage (relative) error . . . . . Student answer. . . . . %

QUESTIONS: *Answer in complete sentences.*

1. What is the experimental result of your determination of the wavelength of the sodium spectrum? . . . . . Student answer. . . . . .

2. What are the possible sources of the error(s) which could account for the difference between the observed and accepted values for the wavelength of the sodium spectrum? . . . . . Errors in apparatus setting, measurement of distances, observational errors, calculations. . . . . .

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Experiment 8

NAME . . . . . PERIOD . . . . . DATE . . . . . .

INSTRUCTOR'S COMMENTS . . . . . .

Covalent Molecules

PURPOSE: To construct models of molecules of covalent substances in order to show how their shapes and structure are related to their polarity.

MATERIALS: Sargent Molecular Model Set or Johnglass Student Molecular Models kit.

INTRODUCTION: A covalent bond consists of a pair of shared electrons. Such a pair of electrons is shared equally between identical atoms producing a bond which is pure (non-polar) covalent. The unequal sharing of the electron pair between two different atoms produces a bond which is more or less polar. The polarity of the bond may be described in terms of its percentage of ionic character. This depends on the electronegativity difference between the two bonded atoms. The electronegativity of an atom (element) is a measure of its tendency to attract the electrons forming a bond between it and another atom.

Molecules of covalent substances may be nonpolar or polar. A diatomic molecule of an elementary gas is nonpolar. A diatomic molecule of a compound is linear (straight line) and may be polar. More complex compounds have molecules which may be polar or non-polar depending on the symmetry of the bonds to the central atom and consequently on the symmetry of the molecular structure. A molecular structure, even though its bonds are polar bonds, will be nonpolar if the bonds are arranged evenly about the central atom. When the arrangement of the polar bonds is unsymmetrical, the resultant molecule is polar and is called a dipole. Representative molecules may be bent (nonlinear) or trigonal pyramids. A dipole acts as if it has a positively charged portion and a negatively charged portion equal in magnitude of charge(s) and separated by some distance.

SUGGESTION: Consult tables of (1) electronegativities and (2) electronegativity difference and percentage of ionic character as needed: See MODERN CHEMISTRY, 1974 edition. Check the Table of Contents of your model set (pasted on the inside of the box cover) with the actual contents. At the end of the experiment, reassemble the kit into the pattern shown on the box cover. Use the long(est) set of spring connectors for multiple bonds.

PROCEDURE: Assemble the first set of seven models and have them checked by the teacher. Fill in the Data Table at the bottom of this page and at the top of the next page. Take these models apart and proceed similarly with the second set.

DATA TABLE

Formula	Electron-dot formula	Bond type (polar or nonpolar)	Shape of molecule (linear, bent, pyramidal, tetrahedral)	Kind of molecule (polar or nonpolar)
Set 1.				
a. H_2	H:H	nonpolar	linear	nonpolar
b. Cl_2	:Cl:Cl:	nonpolar	linear	nonpolar
c. O_2	:O::O:	nonpolar	linear	nonpolar
d. N_2	:N:::N:	nonpolar	linear	nonpolar

Formula	Electron-dot formula	Bond type (polar or nonpolar)	Shape of molecule (linear, bent, pyramidal, tetrahedral)	Kind of molecule (polar or nonpolar)
e. HCl	H:Cl:	polar	linear	polar
f. BrCl	:Br:Cl:	nonpolar	linear	nonpolar
g. HBr	H:Br	polar	linear	polar
Set 2. a. H_2O	H:O: H	polar	bent	polar
b. CO_2	:O::C::O:	polar	linear	nonpolar
c. H_2S	H:S: H	nonpolar	bent	polar
d. NH_3	H :N:H H	polar	pyramidial	polar
e. CH_4	H H:C:H H	nonpolar	tetrahedral	nonpolar
f. CCl_4	:Cl: :Cl:C:Cl: :Cl:	polar	tetrahedral	nonpolar
g. CH_3Cl	H H:C: Cl: H	C-H(nonpolar) C-Cl(polar)	tetrahedral (note that bond distance for C-Cl is different from C-H)	polar

QUESTIONS

1. Calculate the electronegativity difference and percentage of ionic character for each of the following bonds:

	Electronegativity difference	Percentage of ionic character
a. H—O	1.4	39%
b. H—N	0.9	18–19%
c. H—Cl	0.9	18–19%
d. Br—Cl	0.2	1%
e. H—S	0.4	4%
f. H—S	0.4	4%
g. Cl—Cl	0	0%
h. C—O	1.4	22%
i. K—Br	2.0	63%
j. Na—O	2.6	82%

2. Classify each of the following as ionic crystal, polar covalent molecule, or nonpolar covalent molecule.

a. Br_2 . . . nonpolar covalent molecule

b. $MgCl_2$ . . . ionic crystal

c. CCl_4 . . . nonpolar covalent molecule

d. HI . . . polar covalent molecule

e. CO_2 . . . nonpolar covalent molecule

f. H_2O . . . polar covalent molecule

g. N_2 . . . nonpolar covalent molecule

h. $BaBr_2$ . . . ionic crystal

3. Both water and carbon dioxide are triatomic molecules. Explain why one of these is polar and the other is nonpolar. . . . .

The water molecule is unsymmetrical and hence polar. Carbon dioxide has a symmetrical molecule and hence is nonpolar.

Experiment 9

NAME. . . . . PERIOD . . . . . DATE . . . . . .

INSTRUCTOR'S COMMENTS . . . . . .

Determination of Oxygen in Potassium Chlorate

PURPOSE: To determine the mass of oxygen in a sample of potassium chlorate. To gain practice in precision work in the laboratory.

APPARATUS: Asbestos square; balance, platform; balance, sensitive to 0.01 g; beaker, 250 ml; burner and tubing; crucible and cover; forceps; ring, iron; ring stand; tongs; triangle, pipestem; wire gauze.

MATERIAL: Potassium chlorate, C.P.; potassium chloride, C.P.

INTRODUCTION: In this experiment, you will determine the mass of oxygen in potassium. By weighing the potassium chlorate first, then heating it to drive off the oxygen, and then weighing the residue (potassium chloride), you can measure the mass of the oxygen that was in your sample. *Potassium chlorate* melts at 368.4° C and decomposes at 400° C. *Potassium chloride*, the residue, has a melting point of 776° C and sublimes at 1500° C. The method of heating employed in this experiment is based on these properties of the two compounds.

SUGGESTION: Since potassium chlorate tends to adsorb some water vapor from the air, it is suggested that the instructor heat the potassium chlorate overnight in a drying oven at 105° C and cool it in a desiccator before the class assemblies.

By placing the hot crucible on a bright, clean wire gauze without an asbestos center, the time rqeuired for cooling is considerably reduced.

The results of this experiment may also be used to calculate the percentage of oxygen in potassium chlorate. An alternate experiment is Experiment 19(A) Percentage of Water in a Hydrate.

PROCEDURE: 1. Heat a clean, dry procelain crucible and cover on a pipestem triangle supported by a ring stand strongly for 2 minutes. See Fig. F-10. Using clean tongs, transfer the crucible to a clean bright wire gauze, to cool. When cool, weigh the crucible and cover to the nearest 0.01 g. Record the mass in the Data Table. *Do not handle the crucible or cover at any time with the fingers.*

2. Transfer approximately 2 g of potassium chlorate, weighed out on a platform balance on a square of glazed paper, to the weighed crucible. Determine the mass of the crucible, contents, and cover to the nearest 0.01 g and record.

3. Place the crucible on the pipestem triangle, place the cover in position, and heat very *gently* for 2 minutes. Why? At 2-minute intervals gradually increase the temperature until the crucible is heated *strongly*. Keep the crucible covered until near the end of the heating. Reason? After 10 minutes, remove the burner, take off the crucible cover and place it on a clean asbestos square. What is the appearance of the residue? Heat the crucible strongly, without cover, for 2 or 3 minutes longer. Allow the crucible to cool, preferably on the clean wire gauze, and again determine the mass of the crucible, contents, and cover to the nearest 0.01 g.

DATA TABLE

Mass of empty crucible and cover	g
Mass of crucible, contents, and cover before heating	g
Mass of potassium chlorate used	g
Mass of crucible, contents, and cover after heating to constant weight	g
Mass of potassium chloride produced	g
Mass of oxygen driven off	g
Theoretical mass of oxygen in $KClO_3$	g
Error	g
Percentage error	%

Observations: Student answer.

4. To make sure that all of the oxygen has been removed from the potassium chlorate, the crucible and cover should be heated again for about 3 minutes, cooled, and reweighed. If this weighing agrees within 0.01 g of the previous one, you may assume that all of the oxygen had been driven off. Otherwise, a third heating is necessary. This is called *heating to constant weight*, and is done by chemists in all precise work.

Observations: Student answer.

5. After the crucible and cover are cool, place them in a beaker of cold water and apply heat to dissolve the residue remaining in the crucible. What is the name and composition of this residue? Rinse the crucible and cover, and wipe them dry. The solution in the beaker may be discarded.

Observations: potassium chloride, KCl

CALCULATIONS: 1. Calculate the *theoretical* mass of oxygen in your sample of potassium chlorate, expressed to the proper number of significant figures. Show these and subsequent computations in smooth form on a separate paper.
2. Calculate your experimental error and indicate whether your result is low or high by using the correct sign.
3. Calculate your percentage error.
4. The experimental data may also be used to (a) calculate the percentage of oxygen in your sample of potassium chlorate, and (b) to calculate your error after first computing the *theoretical* percentage of oxygen in potassium chlorate.

FURTHER EXPERIMENTATION: Determine the percent of potassium chlorate in a sample of a mixture of potassium chlorate and potassium chloride issued to you by the instructor. Use approximately 2 g of this sample and repeat procedures 1–4. Determine the mass of oxygen driven off. Using the theoretical percentage of oxygen in $KClO_3$, calculate the mass of $KClO_3$ in the mixture. Then calculate the percent of $KClO_3$ in your sample. Report the result to your instructor. Calculate the percentage error based on the data furnished by your instructor.

1. Percentage of $KClO_3$ in sample Student answer.
2. Percentage error. Student answer.

QUESTIONS: *Answer in complete sentences.*
1. What is the experimental result of your determination of the mass of oxygen in the potassium chlorate? Student answer. g
2. What is the theoretical result of your computation of the mass of oxygen calculated from the formula? 39.1% of mass of sample. g
3. What are the possible sources of error which could account for the difference between the experimental and theoretical calculations? Errors in weighing accuracy, limitations of the balance, loss of chemical during heating, failure to remove all the oxygen, contamination of the $KClO_3$.

Experiment 10

NAME. . . . . PERIOD . . . . . DATE . . . . . .

INSTRUCTOR'S COMMENTS . . . . . .

Balancing Chemical Equations

PURPOSE: To show that certain chemical equations may be balanced by the construction and manipulation of molecular models of the reactants and products according to the law of conservation of atoms.

APPARATUS: Apparatus for the electrolysis of water with platinum electrodes (several types are available); beaker, 1000 ml; funnel; graduated cylinder; source of direct current, about 6 volts; test tubes.

MATERIAL: Ball-and-stick model kit (Sargent or Johnglass Molecular Model kit); wooden splints; sulfuric acid, concentrated.

INTRODUCTION: In writing formula equations, each formula must be correctly written. The equation must then be balanced according to the law of conservation of atoms. According to this law, the total number of atoms of each element represented in the reactant formulas must be equal to the total number of atoms of the same element represented in the product formulas. Such balancing is performed by using appropriate *coefficients* in front of each formula. A correctly balanced formula equation furnishes quantitative information about the relative proportions of reactants and products in terms of (a) moles; (b) formula weights; (c) any consistent mass units, such as grams; and (d) for gases only, volumes. The demonstration on the electrolysis of water will show observable experimental results which will be correlated with the use of molecular models in balancing the representative formula equation.

It is suggested that this demonstration be performed, with the aid of students, at the lecture table. The models of reactants and products are to be constructed and manipulated by student teams.

The decomposition of a substance by means of electric energy is called electrolysis. By passing a direct electric current through water, it can be decomposed into the two elementary gases, hydrogen and oxygen, which form this compound.

Pure water is a very poor conductor of electricity. In order to make the liquid a better conductor, a small quantity of sulfuric acid is added. We are interested in (a) the fact that water can be decomposed into oxygen and hydrogen, (b) the relative volumes of these gases which are evolved, and (c) the electrodes at which these gases are discharged.

PROCEDURE: 1. Set up an electrolysis apparatus as shown in Fig. 10-1. Connect the apparatus to one of the following sources of direct current: (a) four dry cells connected in series; (b) a lead storage battery of 6 volts, (c) a low voltage rectifier with 6–8 volt d-c terminals, or (d) a 110-volt d-c outlet with an electric lamp connected in series to regulate the current flowing through the electrolysis apparatus.

2. Slowly, and with stirring, add 100 ml of concentrated sulfuric acid to 600 ml of water in a 1000-ml beaker. With a small funnel, carefully add the acid-water mixture to the apparatus until the tubes are full and a small excess collects in the reservoir. (Open the stopcocks of the tubes to let the air out as the tubes are filled.)

3. When all is ready, close the switch, and observe the activity at each electrode. After the reaction has been going on for a minute or two, open the switch and observe whether the electrolysis will proceed without an external source of energy. Close the switch again and allow the electrolysis to continue until one of the tubes is about two-thirds full of gas.

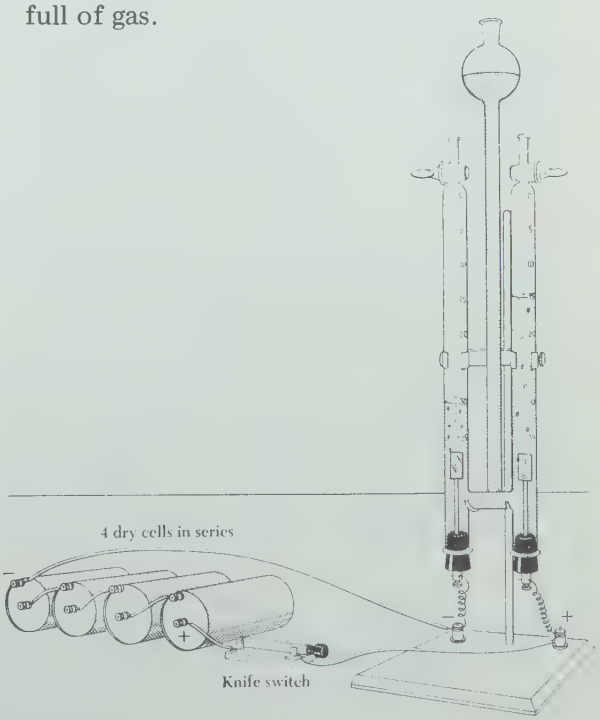

Fig. 10-1

Observations: Student answer.

4. Collect a test tube full of gas from the tube that has the larger volume of gas in the following manner: (a) hold a test tube inverted above the stopcock, (b) open the stopcock, (c) close the stopcock as the tube again fills with liquid. Hold a *blazing* splint over the test tube of gas to identify it. In the same manner, collect a test tube full of gas from the other tube. Thrust a *glowing* splint into this test tube of gas to identify it.

Observations: Student answer.

5. Trace the electric connections to the apparatus to identify the positive and negative electrodes. Which gas collects at the positive electrode? At the negative electrode? Now reverse the electric connections to the apparatus, close the switch, and observe whether the gases collect in the same tubes as before. Result?

Observations: O_2 collects at the positive electrode and H_2 at the negative electrode. Reversing the electric connections results in the same gas-electrode products as previously, i.e. H_2 at negative and O_2 at positive electrodes.

6. Write the word equation for the change which the water undergoes. water → hydrogen + oxygen.

Using correct formulas for the reactant and the products, write the skeleton formula equation for the change which the water undergoes. $H_2O \rightarrow H_2 + O_2$.

Construct the ball and stick models for the reactant and product molecules represented in the skeleton formula equation. (See directions in Experiment 8 if necessary.) Set the models in the same pattern as the formulas appear in the equation. Then construct as many additional models of reactant and products as are necessary to conform to the law of conservation of atoms. Write the complete balanced formula equation which represents the models arrangement. $2H_2O \rightarrow 2H_2(g) + O_2(g)$

7. For each of the skeleton formula equations below construct the necessary number of models of reactants and products and, in each case, in succession, construct enough models and arrange them so that the law of conservation of atoms is followed. Then write in the correct coefficients, corresponding to the number of models appearing in your arrangement, to balance the equation.

$$C + O_2 \rightarrow CO_2$$

$$H_2 + Cl_2 \rightarrow 2HCl$$

$$2NH_3 \rightarrow N_2 + 3H_2$$

$$CH_4 + 2O_2 \rightarrow CO_2 + 2H_2O$$

$$CH_4 + Cl_2 \rightarrow CH_3Cl + HCl$$

QUESTIONS

1. Does the reaction, once started, continue after the source of electricity is disconnected? No. Is electrolysis an exothermic or an endothermic reaction? Endothermic.

2. What relationship do you find between the relative number of molecules represented by the balanced formula equation. (Procedure 6) and the experimentally measured volumes of the gases produced? Identical.

3. What does this seem to indicate about the number of molecules of *any gas* (as represented by the two gaseous products) present in a unit volume at given conditions of temperature and pressure (approximate)?

The number of molecules of any gas present in a unit volume at given conditions of temperature and pressure is the same.

4. The relative volumes of gases *collected in the tubes* are not exactly two to one, although exactly twice the volume of one gas is *set free* compared with the other gas. How do you account for the difference?

Oxygen is more soluble in water than hydrogen. Because of its greater volume, the hydrogen is under greater liquid pressure than the oxygen.

Experiment 11

NAME. . . . . PERIOD . . . . . DATE . . . . .

INSTRUCTOR'S COMMENTS . . . . . .

Mass Relations in a Chemical Change

PURPOSE: To measure the yield of a chemical reaction. To compare the yield with that calculated from the chemical equation.

APPARATUS: Balance, sensitive to 0.01 g; burner and tubing; crucible and cover; ring, iron; ring stand; tongs; triangle, pipestem; bright wire gauze.

MATERIAL: Copper wire, 22-26 gauge; sulfur, powdered.

INTRODUCTION: A chemical equation expresses the relationship between the reactants and products of a chemical reaction in a quantitative sense. By means of a correctly written equation, we may determine, for a particular reaction, the yield that we may expect from a known quantity of reactant.

In this experiment, you will heat a weighed amount of copper with an excess of sulfur to produce a sulfide of copper. The synthesis is carried out in a covered crucible in order to prevent oxygen from combining with the copper. The excess sulfur escapes as a vapor during the heating process. As copper may have an oxidation number of +1 or +2 in combination with sulfur, you will utilize your experimental date to determine the combining proportion of the copper and sulfur atoms and thus the empirical formula of the sulfide produced. This information will enable you to write the chemical equation for the reaction. You may then calculate the theoretical yield of the sulfide from the equation and compare it with the experimental yield of the product.

SUGGESTION: If the laboratory is not equipped with an efficient fume exhaust system, this experiment should be used as a demonstration. The students record the data and make the calculations.

A hot crucible will cool rapidly if removed from the pipestem triangle immediately after removing the flame and placed on a bright, clean wire gauze.

PROCEDURE: 1. Preheat a clean crucible and cover. Recall the technique used in Experiment 9 in this connection. (*See the suggestion above.*) Handle the crucible with clean tongs. Why? When cool, determine the mass of the crucible and cover to the nearest 0.01 g. Record. Wind approximately 2 g of copper wire to form a small compact coil which will fit well down into the crucible (70 cm of 22 gauge wire or 190 cm of 26 gauge wire will do). Find the mass of the copper by the indirect method, that is, by determining the combined mass of the crucible, cover, and copper to the nearest 0.01 g. Record in the Data Table.

2. Add just enough powdered sulfur to cover the copper wire and return the covered crucible to the pipestem triangle (tongs). The reactants should be heated under a hood, or the laboratory should be kept well ventilated if a hood is not available. Heat the covered crucible and contents for about 5 minutes. *Do not remove the crucible cover while the crucible is hot.* Reason? Do you observe any blue flames around the rim of the crucible? Explain. If, after 5 minutes of gentle heating, the blue flames persist, continue heating gently until the flame no longer appears. Now increase the heat intensity gradually until the bottom of the crucible is red hot. Play the flame over the

DATA TABLE

Mass of crucible and cover	g
Mass of crucible, cover, and copper	g
Mass of copper used	g
Mass of crucible, cover, and contents after first heating	g
Constant mass of crucible, cover, and contents	g
Mass of product formed (experimental)	g
Mass of combined sulfur	g
Empirical formula (computed)	Since the compound formed under different conditions is variable, the results will vary. Most students' results approximate the empirical formula Cu_9S_5.
Mass of product computed from equation (theoretical)	g
Experimental error	g
Percentage error	%

sides and top of the crucible to evaporate the last traces of the excess sulfur. Finally heat strongly for 5 minutes. Allow to cool. (Recall the cooling suggestion.)

Observations: Student answers.

3. When cool, remove the cover and see if any sulfur remains on the inside surface of the crucible or cover. If all traces of sulfur are gone, determine the mass of the crucible, cover, and contents to the nearest 0.01 g. Record. Replace the crucible on the pipestem trkangle, add about 1 g of powdered sulfur, replace the cover, and apply heat as before. Allow to cool, inspect for traces of sulfur, and reweigh. If it is within 0.01 g of the last weighing, you may assume a constant mass has been attained. If not, add sulfur, heat, cool, and weigh again. By this method, heat to constant mass. Record this final mass. (Do not discard the product until all computations have been made.) Why? Explain the reason for the second addition of sulfur and reheating.

Observations: You may wish to check the constant mass by adding additional sulfur. The second or-and subsequent additions of sulfur and reheating are necessary in order to make sure that all the copper has reacted.

CALCULATIONS: (on separate paper)

1. Determine the empirical formula of the copper sulfide product. The calculations should indicate either a 1:1 or 2:1 atom ratio.
2. Write the equation for the reaction which yields this product. Find the mass of product as indicated by the amount of copper used and the balanced equation. Show the equation, problem setup, and smooth computation on a separate paper. Enter the result in the Data Table.
3. Determine the experimental error and compute the percentage error. Show the smooth computations and enter the results in the Data Table.

QUESTION: *Answer in complete sentences.*

1. How can you account for your experimental error in this experiment?

(a) Limited accuracy in weighing; (b) use of approximate atomic weight; (c) possible formation of copper(III) oxide (if actual yield is low); (d) failure to evaporate all excess sulfur (if actual yield is high).

Experiment 12

NAME . . . . . PERIOD . . . . . DATE . . . . .

INSTRUCTOR'S COMMENTS . . . . . .

Types of Chemical Reactions

PURPOSE: To show that chemical reactions fall into a rather limited variety of types or categories, as preparation for developing skill in equation writing.

APPARATUS: Asbestos square; beaker (100 ml); burner and tubing; clamp, buret; clamp, test tube; dish, evaporating; forceps; funnel; glass plate; graduated cylinder; medicine dropper; ring, iron; ring stand; stirring rod; test tubes; test tube, Pyrex; wire gauze.

MATERIAL: Filter paper; litmus papers; red and blue; sandpaper, fine grit; calcium, turnings; copper foil; copper(II) sulfate pentahydrate, medium crystals; mercury(II) oxide; sodium hydrogen carbonate; sulfur, powd.; zinc strip, 1 cm × 5 cm; ammonia-water solution, dil. (1:4); hydrochloric acid (3.0 *M*); hydrochloric acid (0.1 *M*); sodium hydroxide (0.1 *M*); aluminum chloride (1.0 *M*); chlorine water; copper(II) sulfate (1.0 *M*); lead nitrate (1.0 *M*); sodium bromide (1.0 *M*); soxium chloride (0.5 *M*); sodium iodide (1.0 *M*); sodium sulfite (1.0 *M*); silver nitrate (0.2 *M*).

ADDITIONAL MATERIAL FOR INSTRUCTOR'S EXPERIMENT: Calcium oxide (lump); zinc, powder.

INTRODUCTION: Three factors must be satisfied in writing formula equations: (a) the equation must represent the facts; (b) the equation must include the symbols and formulas of all elements and compounds used as reactants and formed as products; and (c) the law of conservation of atoms must always be satisfied.

It is advantageous to learn to recognize that reactions fall into the main categories: (1) *composition* reactions, (2) *decomposition* reactions, (3) *replacement* reactions, and (4) *ionic* reactions. In *composition* reactions, two or more substances combine to form a more complex substance. They have the general form A + X → AX. In *decomposition* reactions, the reverse of the first type, a complex substance breaks down to form two or more simpler substances. They have the general form AX → A + X. In *replacement* reactions, one substance is displaced from its compound by another substance. They have the general form A + BX → AX + B or Y + BX → BY + X. In *ionic* reactions of the ion-exchange type, ions in solution combine to form a product that leaves the reaction environment. They may have the general form Al (aq) + Y; (aq) → AY. A sample formula equation for this type has the general form AX + BY → AY + BX. In order for this type of reaction to occur, a product must be formed that separates ions from the reaction environment (the solution) as a solid precipitate or an insoluble or slightly soluble gas, or as a new molecular species (generally water).

SUGGESTIONS: Where the length of the laboratory period prevents each student from doing all parts of this experiment, individual students may be assigned to different sets of procedures. Toward the end of the period they may present their findings to the entire class.

Students are to write the equations for the Instructor's Experiments. They are to use a sample of the experimental product of the reaction between calcium oxide and water in Procedure 1.*c*.

PROCEDURE: 1. *a.* Sandpaper a piece of copper foil (1 cm × 5 cm). Holding one end with forceps, hold it in the outer cone of the Bunsen flame for 1–2 minutes. Let it cool and re-examine it. Name the product, describe it briefly, and complete the representative equation in Data Table 1. . . . . .

The Cu glows forming a black solid.

b. Similarly clean a longer piece of copper foil (1 cm × 25 cm). Place 5 g of sulfur powder in a test tube. Holding the test tube with the buret clamp, heat the sulfur to boiling. Holding the copper foil with the forceps, insert the foil into the sulfur vapor. Result? Examine the product and complete the representative equation in Data Table 1.

The Cu glows in the sulfur vapor forming a black solid.

c. Obtain a one gram sample of the solid product resulting from the Instructor Experiment with calcium oxide and water. Place this in a test tube and add 10 ml of water. Shake the tube for 2 minutes, then

filter. Test the filtrate with red and blue litmus papers. Blue litmus paper turns red in the presence of acids and red litmus paper turns blue in the presence of metallic hydroxides. Name the product. Describe the product briefly, and complete the equation in Data Table 1 which pertains to it.

2. In each part of this procedure, 5 grams of the reactant will be placed in separate test tubes, held with the buret clamp, and heated for three minutes. The appearance of the product is to be observed. Certain products may be identified according to specific directions. Use Data Table 2 to write the complete, balanced equation, and to name the products. Describe their appearance or identification procedures.

a. Heat the sample of mercury(II) oxide. Insert a glowing splint into the upper part of the test tube. Result? Note the appearance of any deposit on the side of the test tube. Record your results.

b. Heat the sample of sodium hydrogen carbonate holding the test tube in a horizontal position. Insert moistened pieces of red and blue litmus paper into the upper part of the test tube. Result? Insert a flaming splint into the upper part of the test tube. Result? Positive results identify carbon dioxide. Note any deposit on the sides of the test tube. Record your results. Retain this test tube and its contents for part 4*e*.

c. Heat the sample of copper(II) sulfate pentahydrate holding the test tube at an angle, mouth downward. What substance appears on the colder portion of the test tube? Note any change in appearance of the solid left in the test tube. Record your results.

3. Follow specific directions for each part of this procedure. Record details in Data Table 3 as in previous procedures.

a. Add 5 ml of hydrochloric acid to a test tube. Drop a cleaned zinc strip into the acid. After one minute, bring a flaming splint to the mouth of the test tube. Result? Fill the test tube with water and pour off the liquid in the test tube.

b. Add 5 ml of the copper(II) sulfate solution to a test tube. Drop a cleaned zinc strip into the solution. Permit the strip to remain in the solution for 10 minutes. Note the substance deposited on the zinc strip and any change in the color of the solution.

c. Repeat procedure (*b*) using 5 ml of silver nitrate solution into which a piece of copper foil is dropped. Make similar observations.

d. Add 5 ml of the sodium bromide solution to a test tube. Add 1 ml of chlorine water to this solution and note any change in the color of the solution. (Elementary bromine in water solution has a red-brown color.)

e. Add 5 ml of water to a test tube. Drop a clean calcium turning into the water. Result? Immediately bring a flaming splint to the test tube mouth. Result? Insert pieces of red and blue litmus paper into the resultant solution. Result?

4. Follow specific directions for each part of this procedure. Record details in Data Table 4 as in previous procedures.

a. Add 1 ml of silver nitrate solution to 5 ml of sodium chloride solution in a test tube. Use the Table of Solubility of Salts on page 281 of MODERN CHEMISTRY, if necessary, to identify the products. Record your results.

b. To 5 ml of sodium sulfite solution add 1 ml of hydrochloric acid. Result? Test for the odor of a product by using the procedure of Fig. F-5. Result? Insert moistened pieces of red and blue litmus paper into the upper part of the test tube. Record your results.

DATA TABLE 1

Complete, balanced equation	Name of product	Brief description of product
$Zn + S \rightarrow$	ZnS zinc sulfide	white powder
$2Cu + O_2 \rightarrow$	2 CuO copper(II) oxide	black solid
$2Cu + S \rightarrow$	Cu_2S (or CuS) copper(I) sulfide	black solid
$CaO + H_2O \rightarrow$	$Ca(OH)_2$ calcium hydroxide	white powder

DATA TABLE 2

Complete, balanced equation	Products	Identification or appearance of products
$2HgO \rightarrow 2Hg + O_2(g)$	Hg, O_2	Silvery mirror, (Hg), glowing splint test (O_2).
$2NaHCO_3 \rightarrow Na_2CO_3 + H_2O + CO_2(g)$	(see equation)	H_2O condenses on test tube, CO_2 extinguishes flaming splint, Na_2CO_3 reacts with HCl.
$CuSO_4 \cdot 5H_2O \rightarrow CuSO_4 + 5H_2O$	(see equation)	H_2O condenses and runs out of test tube; white powdered, anhydrous $CuSO_4$.

DATA TABLE 3

Complete, balanced equation	Products	Identification or appearance of products
$Zn + 2HCl \rightarrow ZnCl_2 + H_2(g)$	(see equation)	Blazing splint ignites H_2 with a bark.
$Zn + CuSO_4 \rightarrow Cu + ZnSO_4$	(see equation)	First a black deposit which turns red (Cu) forms on the Zn; $CuSO_4$ solution becomes colorless $ZnSO_4$ (eventually.
$Cu + 2AgNO_3 \rightarrow 2Ag + Cu(NO_3)_2$	(see equation)	Needle shaped crystals of silver are deposited; colorless $AgNO_3$ turns to blue $Cu(NO_3)_2$.
$2NaBr + Cl_2 \rightarrow 2NaCl + Br_2$	(see equation)	Red-brown solution of Br_2.
$Ca + 2H_2O \rightarrow Ca(OH)_2 + H_2$	(see equation)	Blazing splint ignites H_2 with a bark. Litmus turns blue in solution.

DATA TABLE 4

Complete, balanced equation	Products	Identification or appearance of products
$AgNO_3 + NaCl \rightarrow NaNO_3 + AgCl(s)$	see equation	White precipitate of AgCl.
$Na_2SO_3 + 2HCl \rightarrow 2NaCl + H_2O + SO_2(g)$	see equation	Choking odor of SO_2 which turns blue litmus to red (H_2SO_3).
$AlCl_3 + 3NH_3 + 3H_2O \rightarrow 3NH_4Cl + Al(OH)_3(s)$	see equation	White, gelatinous precipitate of $Al(OH)_3$.
$NaOH + HCl \rightarrow NaCl + H_2O$	see equation	White crystals of NaCl.
$Na_2CO_3 + 2HCl \rightarrow 2NaCl + H_2O + CO_2(g)$	see equation	Blue litmus turns red and glowing splint extinguished indicating production of CO_2.
$2NaI + Pb(NO_3)_2 \rightarrow 2NaNO_3 + PbS(s)$	see equation	A yellow precipitate of lead(II) iodide.

c. To 5 ml of aluminum chloride solution add 1 ml of ammonia-water solution. Result? Refer to the Table of Solubility of Salts in the text, if necessary, to identify the products.

d. Place 5 ml of hydrochloric acid (0.1 *M*) in the beaker. Add 4 ml of sodium hydroxide solution (0.1 *M*) to the acid with stirring. Place one drop of the resultant solution in separate pieces of red and blue litmus paper (on a clean glass plate over white paper). Result? Continue to add the sodium hydroxide solution dropwise to the beaker contents (with stirring) until the test litmus papers undergo no change (red stays red and blue stays blue). Evaporate 5 ml of the fluid (in the beaker) in an evaporating dish over a wire gauze with asbestos center, using a very low flame near the end of the process. When only a few drops of liquid remain, remove the burner and allow the residual heat to evaporate the last few drops. Allow the dish to cool and identify the residue.

e. Add 2 ml of hydrochloric acid to the residue left in the test tube of part 2*b*. Insert moistened pieces of red and blue litmus paper into the upper part of the test tube. Result? Insert a flaming splint into the upper part of the test tube. Result?

f. Add 5 ml of sodium iodide solution to a test tube. Add 5 ml of lead nitrate solution. Result? Use the Table of Solubility of Salts in the text to identify the product(s).

g. Complete Data Table 4.

INSTRUCTOR EXPERIMENT: 1. Mix 2 g of zinc powder with 4 g of sulfur powder in a mortar. Make a small cone of the mixture on the asbestos square. Holding the Bunsen burner by the base, play the tip of the flame on the tip of the cone of the mixture.

2. Place approximately 50–60 g of fresh lump calcium oxide in a beaker. Add approximately 18 g of water. Have students observe the result. Dispense the powder resultant to the class for procedure 1.*c*.

QUESTIONS: The terms *type of reactant* or *type of product* mean element(s) and/or compound(s).

1. What type(s) of reactant(s) typify composition reactions? Elements. Compounds.

What type(s) of product(s) typify composition reactions? Compounds.

2. What type(s) of reactant(s) typify decomposition reactions? A compound.

What type(s) of product(s) typify decomposition reactions? Elements, compounds.

3. What type(s) of reactant(s) typify replacement reactions? One element and one compound.

What type(s) of product(s) typify replacement reactions? A different element and a different compound.

4. What type(s) of reactant(s) typify ion-exchange reactions? Usually two compounds.

What type(s) of product(s) typify ion-exchange reactions? Usually two compounds different from the reactants.

Experiment 13

NAME . . . . . PERIOD . . . . . DATE . . . . .

INSTRUCTOR'S COMMENTS . . . . . .

Gas Laws

PURPOSE: To show how the volume of a fixed mass of a gas (air) varies (*a*) with changes in pressure at constant temperature, and (*b*) with changes in temperature at constant pressure.

APPARATUS: Barometer, mercurial; beaker, 600 ml; burner and tubing capillary tube, 40 cm length (either No. 1303 or No. 1303A, Welch); clamp, buret; Charles law tube, mounted (No. 1674A, Welch). Welch apparatus is recommended but details as to preparation and use of alternate glassware are described in references in the Teacher Edition of the text; (2) hydrometers or graduated cylinders, 500 ml (one polypropylene); meter stick; protractor; ring, iron; ring stand; thermometer; tongs; beaker; wire gauze.

MATERIAL: Mercury.

INTRODUCTION: According to Boyle's law, the volume of a certain amount of dry gas is inversely proportional to the pressure, provided the temperature remains constant. This law may be stated mathematically as $\frac{V}{V'} = \frac{p'}{p}$ where V is the original volume, V' the new volume, p the original pressure, an p' the new pressure. This law may also be stated mathematically as $pV = k$ (a constant) where p and V represent corresponding sets of values for the fixed mass of gas.

According to Charles' law, the volume of a certain amount of dry gas varies directly with the Kelvin temperature, provided the pressure remains constant. This law may be stated mathematically as $\frac{V}{V'} = \frac{T}{T'}$ where V is the original volume, V' the new volume, T the original *Kelvin* temperature, and T' the new *Kelvin* temperature. This law may also be stated mathematically as $\frac{V}{T} = k$ (a constant) where V and T represent corresponding sets of values for the fixed mass of gas.

PROCEDURE: 1. *Volume variation with pressure changes.* Set up the capillary tube containing approximately 20 cm of mercury in the vertical position, Fig. 13-1. Measure the length of the enclosed air column to the nearest millimeter. Now measure the height of the upper and lower levels of the mercury column above the table top. Enter these values in the Data Table for the vertical position, 90 degrees.

DATA TABLE

Position (angle) of capillary tube	Vertical 90°	60°	30°	Horizontal 0°	Inverted
V (length of air column, mm)					
Height of upper level of mercury, mm					
Height of lower level of mercury, mm					
p (applied pressure, mm of mercury)				Atmospheric = mm Hg	
pV					

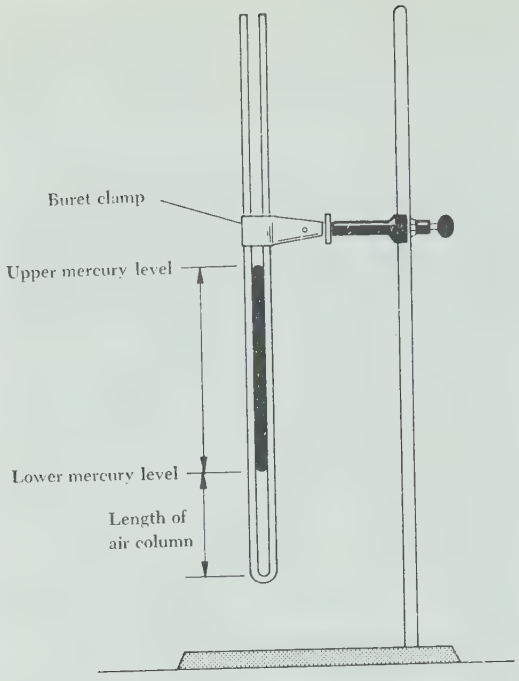

Fig. 13-1

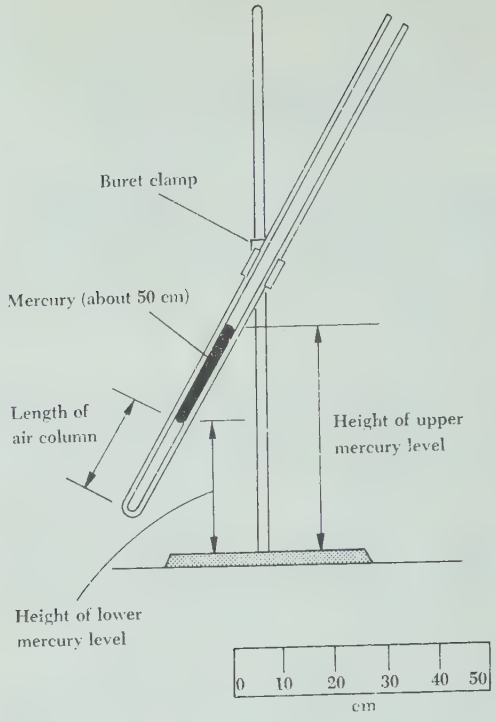

Fig. 13-2

Rotate the tube to the horizontal position. Measure the length of the enclosed air column. Record this quantity in the Data Table for the horizontal position where the angle is 0 degrees. In this position, the pressure on the enclosed air column is equal to atmospheric pressure. Read the atmospheric pressure on the lecture table barometer and enter it as the pressure for the horizontal position.

Rotate the capillary tube until it makes an angle of 60 degrees with the horizontal, using the protractor to check the angle. Measure the height of the air column and the upper and lower levels of the mercury column as shown in Fig. 13-2. Record the measurements in the Data Table. Change the position of the tube to a 30 degree angle and repeat the measurements made for the 60 degree position.

Now place the capillary tube in the inverted position with the open end of the tube facing downward. In this position, the enclosed air is under less than atmospheric pressure, the applied pressure being equal to the *difference* between atmospheric pressure and the height of the mercury column. Measure the height of the air column and the upper and lower levels of the mercury column. Record these quantities in the Data Table.

Calculate the applied pressure for the vertical position (90 degrees) and for the 60 and 30 degree positions respectively by adding the measured height of the mercury column to the atmospheric pressure. Enter these quantities in the Data Table.

Calculate the pV product for each position of the capillary tube and enter the results in the Data Table.

On a piece of graph paper plot a graph of volume versus pressure for the readings obtained. Use the horizontal axis for p values (the applied pressure in mm of mercury) and the vertical axis for V values (the length of the enclosed air column in mm). What is the shape of this graph? What mathematical relationship does such a curve represent?

2. *Volume variation with temperature.* In this part of the experiment you will use the Charles' law tube assembly (Fig. 13-3) which consists of a 40 cm capillary tube containing a column of dry air confined by a short thread of mercury. Set up this tube in a vertical position, clamp it to the ring stand. Measure the length of the enclosed air column and the room temperature. Record these measurements in the Data Table. (p. 132)

Fill a cylinder (hydrometer jar) almost completely with a mixture of ice and water and take the temperature of the mixture to the nearest degree. Now insert the capillary tube assembly as shown in Fig. 13-4. Initially, the bottom of the mercury thread should be at the same level as the top of an ice-water mixture. Cooling the enclosed air column results in some contraction so that the mercury thread will also be pushed

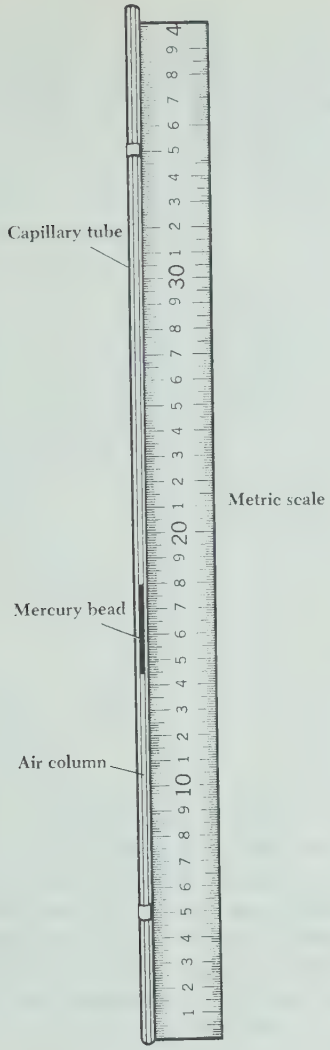

Fig. 13-3

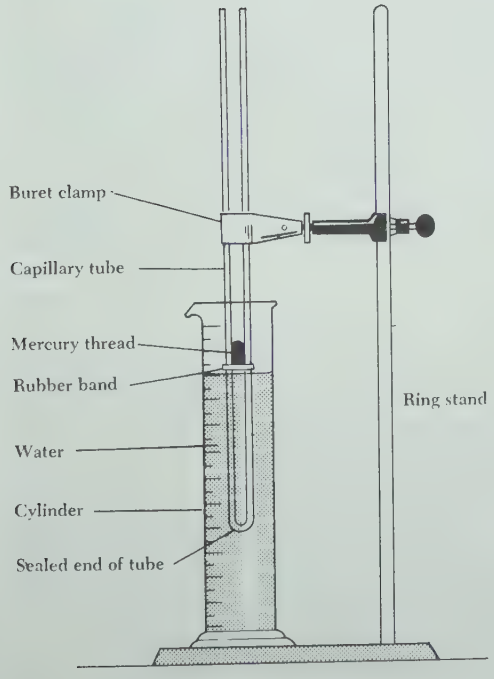

Fig. 13-4

downward by the combined pressure of the atmosphere and the pressure due to the mercury thread. After the mercury thread has stopped moving, raise the capillary tube assembly so that once again the bottom of the mercury thread and the top of the ice-water mixture are at the same level. Measure the length of the air column and record this measurement as well as the temperature of the ice-water mixture in the Data Table.

Remove the ice-water mixture and replace with approximately the same volume of hot water which has been heated in a beaker. The temperature of the hot water should be approximately 40° C. For hot water, use plastic hydrometer or graduated cylinder. Repeat the general procedure in the preceding paragraph and record the length of the air column and the temperature of the hot water in the Data Table. Reheat the water in the beaker to about 60 degrees, pour it into the cylinder, and again measure length of air column and the temperature. Record these quantities in the Data Table. Reheat the water in the beaker to about 80 degrees and repeat the previous procedure.

(*Optional:* Take a final set of measurements for water boiling in the beaker. Record the measurements in the Data Table.

On another sheet of graph paper, use the horizontal axis for Celsius temperatures for the range −300° C to 100° C. The vertical axis is used for recording volumes of air. Actually this corresponds to length of the air column in millimeters. Use an appropriate scale starting with 0 mm so that the measured volumes (lengths of air column) fit on the graph paper. Plot the values you found on the graph and construct the graph using a solid line for these values. Extend this graph to the left, using a dashed line, until it intersects the temperature axis. At what Celsius temperature would the extrapolated volume of the gas be zero?

Convert the Celsius temperatures in the Data Table to Kelvin temperatures and record in the appropriate column in the Data Table. Calculate values for $\frac{V}{T}$ from the recorded lengths of air column and Kelvin temperatures. Enter these values in the Data Table.

Convert the lowest possible Celsius temperature on your graph to the Kelvin scale. This is the temperature at which the theoretical volume of the gas would be zero. The value for Absolute zero on the Kelvin scale is zero, and on the Celsius scale it is nearly −273° C. What is the difference between your Celsius value for Absolute zero and −273° C? Calculate the experimental and percentage errors for this experiment.

DATA TABLE

Gas Volume, V (length of column, mm)	Thermometer Temperature (°C)	Temperature, T (°K)	$\frac{V}{T}$
	(ice-water mixture)		
	(room temperature)		
	(approximately 40°)		
	(approximately 60°)		
	(approximately 80°)		
	(boiling water)		

Experimental error is the difference between your value and the accepted value (−273°C). Percentage error is the experimental error divided by the accepted value and converted to a percentage.

Substitute the Kelvin temperatures for the Celsius temperatures on your graph. Assuming that your extrapolated Celsius temperature corresponds to Absolute zero, what mathematical relationship does the shape of your graph indicate between V, volume of a gas held at constant pressure and T, Kelvin temperature?

Observations: Student answers.

FURTHER EXPERIMENTATION: Additional data for temperature variations may be obtained by using the mercury tube in (a) boiling water or steam, (b) ice-NaCl(s), (c) Dry ice-acetone. An oil drop may be substituted for the mercury for the low-temperature readings since the oil solidifies and makes the measurement easier.

Experiment 14

NAME . . . . . PERIOD . . . . . DATE . . . . . .
INSTRUCTOR'S COMMENTS . . . . . .

Percentage of Oxygen in Air

PURPOSE: To find the percentage of oxygen in a sample of air.

APPARATUS: *Phosphorus method:* battery jar; clamp, buret; eudiometer, 50 ml; metric ruler; ring stand; thermometer. *Pyrogallic acid method:* balance, platform; battery jar; eudiometer, 50 ml; graduated cylinder; rubber stopper, solid No. 0.

MATERIAL: *Phosphorus method:* copper wire, bare, No. 16, 60-cm long; phosphorus, white. *Pyrogallic acid method:* Pyrogallic acid; solution of sodium hydroxide (1.0 *M*).

INTRODUCTION: If a volume of air is measured accurately and then the oxygen removed by chemical means, the percentage of oxygen in the original sample may be computed easily. White phosphorus unites readily with oxygen and forms a white smoke of diphosphorus pentoxide. The white smoke gradually settles and dissolves in the water. The volume of gas remaining, subtracted from the original volume of air, gives the volume of oxygen removed.

An alternate method that gives quicker results is to add a mixture of pyrogallic acid and sodium hydroxide solution to a measured volume of air contained in a stoppered eudiometer. This mixture rapidly unites with the oxygen in the air. The stoppered tube is inverted a few times to mix the chemicals thoroughly with the enclosed air. The stoppered end is then thrust under water in a battery jar. As soon as the stopper is removed, water is forced in to take the place of the removed oxygen. The volume of gas remaining in the tube, subtracted from the volume of air used, gives the volume of oxygen.

PROCEDURE: 1. *Phosphorus method.* Add sufficient water to a 50-ml eudiometer so that 40 to 50 ml of air will be enclosed when the tube is inverted in a battery jar of water. It is a good plan to fill several large vessels with water the day before the experiment, so that the water will be at room temperature. Determine accurately the volume of air enclosed in the eudiometer. Measure the difference in water levels, if any, in millimeters. Record the temperature of the water and also the barometer reading in the room. Compute the volume of dry air enclosed at STP.

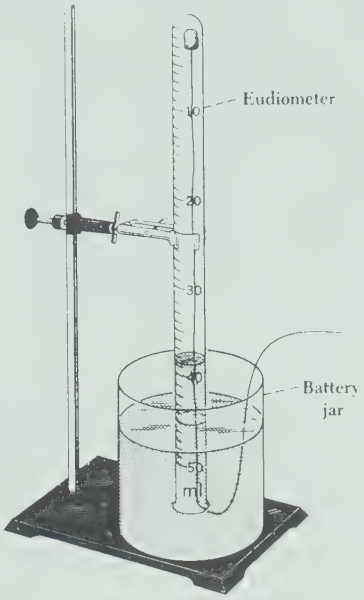

Fig. 14-1

Cut a stick of phosphorus *under water* so as to get a piece with freshly cut surfaces the size of a small bean. **CAUTION:** *Do not handle phosphorus with the fingers.* Thrust one end of a flexible, bare, copper wire, about 60 cm long, into the piece of phosphorus *white it is still under water.* Now push the wire, with the phosphorus on the end, up into the eudiometer to within several centimeters of the top. See Fig. 14-1. Observe the reaction which begins at once. Allow at least 24 hours for the reaction to be completed.

INITIAL DATA

Volume of air enclosed in eudiometer	ml
Difference in water levels	mm
Equivalent difference in levels in mm of mercury	mm
Temperature	°C
Barometer reading	mm
Water vapor pressure	mm
Volume of dry air enclosed at STP	ml

FINAL DATA

Volume of gas remaining	ml
Difference in water levels	mm
Equivalent difference in levels in mm of mercury	mm
Temperature	°C
Barometer reading	mm
Water vapor pressure	mm
Volume of gas remaining at STP	ml
Volume of dry O_2 removed	ml
Percentage of O_2 in sample of air taken	%
Accepted percentage of O_2 in air	%
Percentage error	%

When the white smoke has disappeared, remove the copper wire, measure the difference in levels, and read the volume of the gas remaining. Record the temperature of the water and the barometer reading. Compute the volume of dry gas remaining at STP.

2. *Pyrogallic acid method.* Exactly 50 ml of air will be taken as the sample in this method. That volume of sodium hydroxide solution will be used which is just equal to the volume between the 50-ml mark on the eudiometer tube and the bottom of the rubber stopper which is used to close it. See Fig. 14-2. This volume is easily determined as follows: (1) Fill the tube with water to the 50-ml mark. (2) Insert the rubber stopper. (3) Invert the tube and read from the graduations the volume of air which *was* between the 50-ml mark and the stopper before the tube was inverted. This is the volume of sodium hydroxide solution to use with the tube. (4) Empty the water out of the tube.

Add 0.5 g of pyrogallic acid powder to the tube. Accurately measure in a graduated cylinder the required volume of sodium hydroxide solution and pour it into the tube. Quickly close the tube with the rubber stopper. Invert the tube a few times to mix the contents. Place the stoppered end of the tube under water in a battery jar and then remove the stopper. Note how the water is forced in to replace the oxygen. Allow the tube to cool for a few minutes. Now raise or lower the tube so that the level of water in the tube is the same as that in the battery jar. Read the volume of gas remaining in the tube. It is not necessary to correct the gas to STP in this method because the operations are all done within a few minutes. The volume of gas remaining, subtracted from the original volume of air, 50 ml, gives the volume of oxygen. The volume of oxygen divided by the volume of the sample taken and multiplied by 100% will give the percentage of oxygen in air. Repeat the experiment several times until consistent results are obtained.

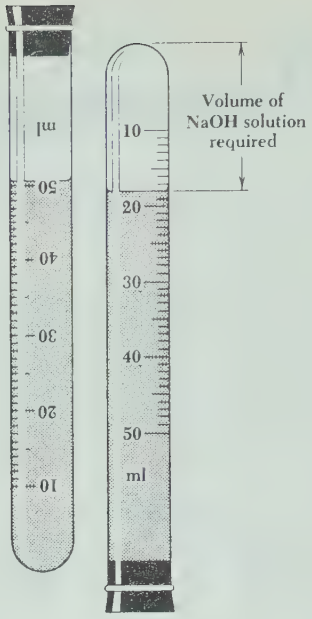

Fig. 14-2

DATA TABLE

	1	2	3
Volume of gas remaining	ml	ml	ml
Volume of oxygen removed	ml	ml	ml
Percentage of oxygen	%	%	%
Accepted percentage of oxygen in air	%	%	%
Percentage error	%	%	%

Experiment 15

NAME . . . . . PERIOD . . . . . DATE . . . . . .

INSTRUCTOR'S COMMENTS . . . . . .

Replacement of Hydrogen by a Metal

PURPOSE: To determine the mass of magnesium needed to replace one gram of hydrogen from a compound.

APPARATUS: Balance, analytical; battery jar, 1 qt; clamp, buret; eudiometer, 50 ml; graduated cylinder; ring stand; thermometer.

MATERIAL: Magnesium ribbon, untarnished; thread; hydrochloric acid, concentrated.

SUGGESTIONS: To save time in weighing, the instructor may weigh a bright, untarnished piece of magnesium ribbon a meter or more in length (allow approximately 6 cm per student). An analytical balance should be used and the mass should be determined to the nearest 0.001 g. Assuming the ribbon to be uniform, the length of sections having a mass from 0.040 to 0.045 g may be determined. Such an amount of magnesium will liberate 40 to 45 ml of hydrogen gas.

INTRODUCTION: The law of definite composition holds true for all compounds. Therefore, it is possible to find out how many grams of one element are needed to unite with *one gram* of another element, hydrogen for example. As a rule, metals do not combine readily with hydrogen, but they do displace hydrogen from compounds, such as hydrochloric acid. Magnesium will react with hydrochloric acid to liberate hydrogen gas. In this experiment, we shall undertake to find out just how many grams of magnesium are required to replace one gram of hydrogen from hydrochloric acid.

Time permitting, your instructor will issue a second piece of magnesium whose mass you are to determine from the volume of hydrogen liberated.

PROCEDURE: 1. Half fill a battery jar with water. If possible, use water that has adjusted to room temperature. Roll a length of magnesium ribbon of known mass into a loose coil. Tie it with one end of a piece of thread, approximately 25 cm in length, in such manner that all the loops of the coil are tied together. Pour 5 ml of concentrated hydrochloric acid into the gas measuring tube, or eudiometer. Add enough water to the eudiometer to fill it completely, being careful not to mix the water and the acid. Reason? Lower the magnesium coil into the water in the gas measuring tube to a depth of about five centimeters. Close the tube with your thumb so that the thread is held firmly against the edge of the tube. Taking care that no air enters, invert the eudiometer in the battery jar and allow it to rest against the bottom to hold the thread. It may be clamped in this position on the ring stand, as shown in Fig. 15-1. The acid flows down the tube (why?) and reacts with the magnesium. Is the acid now concentrated or dilute?

Observations: Student answer. . . . . .

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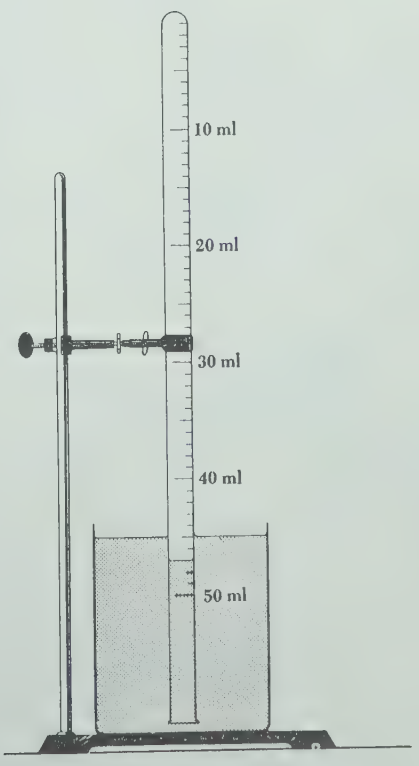

Fig. 15-1

2. When the magnesium has disappeared entirely and the reaction has stopped, adjust the tube until the liquid levels inside and outside are the same. Add water to the battery jar if necessary, using water at room temperature. (If this adjustment is not possible with the apparatus you are using, what computation can you carry out that will yield the same result?) Read as accurately as possible the volume of hydrogen liberated. Take the temperature of the water in the battery jar and assume this to be the temperature of the hydrogen gas collected. Why is this a reasonable assumption? Take a barometer reading from the laboratory barometer. Record these data in the Data Table.

CALCULATIONS: 1. From the table, Pressure of Water Vapor, in the textbook, determine the correction that must be made in the barometer reading to yield the partial pressure of dry hydrogen gas in the eudiometer. Then calculate the volume of the hydrogen gas under standard conditions of temperature and pressure. Enter the result in the Data Table.

2. Calculate the mass of hydrogen liberated, recognizing that this mass of hydrogen bears the same relationship to the volume of hydrogen collected at STP as the g-mol. wt. of hydrogen bears to the g-mol. vol. of the gas. Enter the result in the Data Table.

DATA TABLE

Mass of Mg used	g
Volume of H_2 collected (at laboratory conditions)	ml
Temperature of H_2 collected	°C
Barometer reading	mm
Vapor pressure of water at observed temperature	mm
Pressure of the dry H_2 collected	mm
Volume of H_2 (corrected to STP)	ml
Mass of H_2 liberated	g
Mass of Mg replacing 1 g of H_2 (experimental)	g
Theoretical mass of Mg replacing 1 g of H_2	g
Experimental error	g
Percentage error	%

3. From the mass of magnesium used and the mass of hydrogen evolved, calculate the mass of magnesium needed to replace 1 g of hydrogen. From the balanced equation for this reaction, determine the mass of magnesium theoretically required to replace 1 g of hydrogen. Compute your percentage error. Enter the results in the Data Table.

FURTHER EXPERIMENTATION: Repeat Procedure 1 using the unweighed sample of magnesium ribbon issued to you by the instructor. From the volume of hydrogen collected at laboratory conditions, calculate the mass of the hydrogen liberated according to the directions appearing under CALCULATIONS 1 and 2. Then calculate the mass of magnesium that would theoretically replace this mass of hydrogen. Report this mass to your instructor. Calculate your percentage error on the basis of the data he furnishes regarding the mass of the magnesium sample issued.

DATA TABLE

Reported mass of Mg	g
True mass of Mg	g
Experimental error	g
Percentage error	%

QUESTIONS: *Answer in complete sentences.*

1. Why is it necessary to make a water-vapor pressure correction of the barometer reading in this experiment? A gas collected over water has a pressure which is the sum of the partial pressure of the dry gas and the partial pressure of the water vapor present.

2. What are the possible sources of the experimental error you have shown? (a) Inaccuracy in measuring volume of H_2. (b) error in adjusting gas to atmospheric pressure. (c) permitting air to enter eudiometer, (d) temperature difference between gas and water.

EQUATION

$$Mg + 2HCl \rightarrow MgCl_2 + H_2(g)$$

Experiment 16

NAME . . . . . PERIOD . . . . . DATE . . . . . .

INSTRUCTOR'S COMMENTS . . . . . .

Molar Volume of a Gas

PURPOSE: To determine the molar volume of a gas experimentally.

APPARATUS: Balance, sensitive to 0.01 g; beaker, 400 ml; burner and tubing; clamp, buret; flask, Florence, 500 ml; glass bend, 8 × 8 cm; glass tubing, 6 mm; graduated cylinder; pinch clamp; ring stand; rubber connector; rubber tubing, connector; rubber stoppers, 1-hole No. 4, 2-hole No. 5; test tube, Pyrex; thermometer; wing top.

MATERIAL: Manganese dioxide, C.P.; potassium chlorate, C.P.

INTRODUCTION: The gram-molecular volume, or *molar volume*, of a gas is the volume of 1 mole (1 g-mol. wt.) of the gas at standard conditions of temperature and pressure. A mole of any gas will have a volume equal to that of a mole of any other gas measured under the same conditions. You can see that this is true as a consequence of two important principles which you have learned. First, one mole of any molecular substance consists of the same number of molecules as one mole of any other molecular substance. Second, equal volumes of all gases consist of the same number of molecules when measured under the same conditions of temperature and pressure.

In this experiment, you will collect a known mass of oxygen by water displacement under known conditions of temperature and pressure. You will measure the volume of water displaced as an indication of the volume of oxygen collected. The volume of this known mass of oxygen can then be corrected to standard conditions from which the volume of a mole of the gas may be found.

SUGGESTION: Since potassium chlorate tends to adsorb some water vapor from the air, it is suggested that the instructor heat the potassium chlorate overnight in a drying oven at 105° C and cool it in a dessicator before the class assembles.

By placing the hot crucible on a bright, clean wire gauze without an asbestos center, the time required for cooling is considerably reduced.

If balances, sensitive to 0.001 g are available, better results are obtained.

If students are not permitted to use potassium chlorate, the experiment may be used as a demonstration. An alternate to this experiment using Dry Ice is described in *Chemistry*, 43 (1970—Jan), Lab Bench: "*Molar Volume: A Safe Method.*"

PROCEDURE: 1. Heat about 1 g of powdered manganese dioxide in the Pyrex test tube momentarily to drive off moisture. While the test tube is cooling, assemble the two glass tubing-rubber stopper connections as shown in Fig. 16-1. Add about 1.2 g of dry potassium chlorate to the test tube and mix thoroughly with the manganese dioxide. Insert the dry stopper assembly, determine the mass of the tube and contents to the nearest 0.01 g, and record. Clamp the tube on the ring stand with the stoppered end tilted upward at a small angle.

2. Completely fill the 500-ml flask with water and assemble as in Fig. 16-1. Add water to the beaker to a depth of 1 or 2 cm. Open the pinch clamp and blow on the generator end of the rubber connector to force water out of the flask and over into the beaker so as to displace the air in the interconnecting tube. Keeping the end of the glass tube submerged in the water in the beaker, raise the beaker until the two water levels are the same. (The clamp is open during this operation.) How does this affect the pressure inside the flask? Move the beaker up or down so as to bring the level of the water in the flask into the neck but below the end of the short bend. Reset the clamp while the beaker is in this position, empty the beaker, and wipe it dry. Attach the rubber connector to the oxygen generator and release the pinch clamp. If drops of water run out of the rubber tube for no more than a few seconds, the system is airtight. Put the *dry* beaker back in place.

3. With the wing top on the burner and holding the burner base in your hand, heat the potassium chlorate gently (BE SURE THE PINCH CLAMP IS OPEN). Regulate the heating so as to maintain a moderate flow of oxygen. Avoid excessive heating as shown by the formation of a white cloud in the generating tube. *Under no circumstances allow the water level in the flask to drop below the end of the long glass tube.* Discontinue the heating when the beaker is nearly full. Leave the apparatus undis-

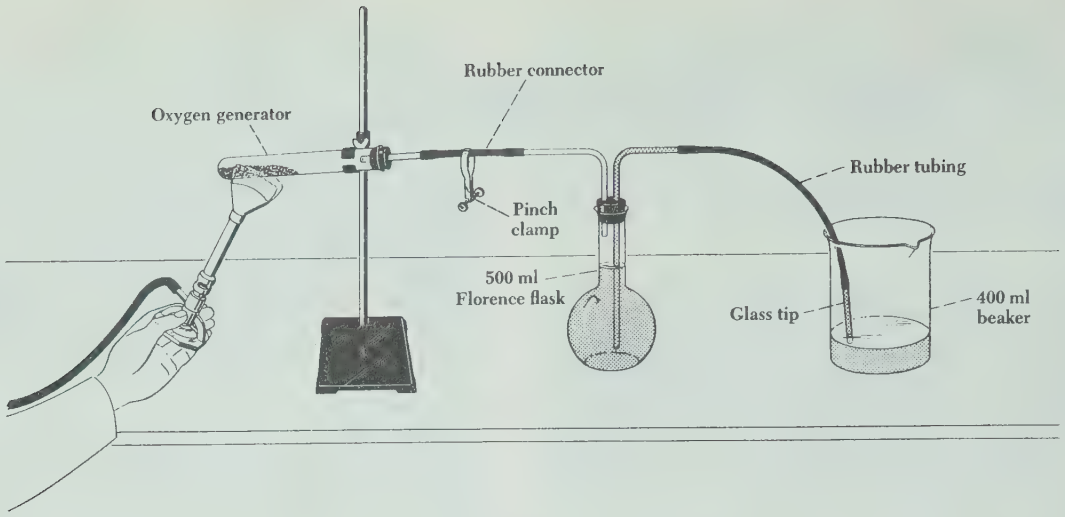

Fig. 16-1

turbed until the generator has cooled to room temperature. Why?

4. While the generator is cooling, obtain the barometer reading. Read the temperature of the water in the beaker. We may assume this to be the temperature of the oxygen. Record.

5. When the generator is at room temperature, make the levels of water in the flask and beaker equal by lowering the beaker. Avoid handling the flask during this manipulation. Why? Be sure that the end of the tube remains below the surface of the water in the beaker. Reset the pinch clamp while the identical levels are maintained, then remove the beaker of water. Carefully measure the volume of water in the beaker. This volume is equal to the volume of oxygen collected in the flask. Record. Disconnect the generator tube containing the residue and weigh, with stopper assembly in place, to the nearest 0.01 g. Record.

CALCULATIONS: 1. The oxygen collected in the flask will be saturated with water vapor as determined by the temperature of the system. Thus the pressure of the gas is due to the partial pressures of the dry oxygen and the water vapor. Determine the partial pressure of the dry oxygen gas produced in the generator and enter in the Data Table.

2. You now have the volume, temperature, and pressure of the known mass of oxygen collected. Determine the volume occupied by this mass of oxygen under standard conditions of temperature and pressure and enter in the Data Table.

3. Since one mole of oxygen is 32 g, the volume occupied by the mass of oxygen at STP will represent the *molar volume* of the gas. Calculate this value, using your known mass of oxygen and its volume at STP. Enter your *experimental* molar volume, in liters, in the Data Table.

4. Taking the accepted molar volume of any gas as 22.4 liters, determine your percentage error and enter the result in the Data Table.

DATA TABLE

Mass of generator before heating . .	g
Mass of generator after heating . . .	g
Mass of oxygen collected	g
Barometer reading	mm
Water vapor pressure at temperature of O_2	mm
Partial pressure of O_2 collected . . .	mm
Temperature of O_2 collected	°C
Volume of O_2 collected	ml
Volume of O_2 reduced to STP . . .	ml
Molar volume of O_2 (exptl.)	liter
Molar volume of any gas (accepted value)	liter
Experimental error	liter
Percentage error	%

NAME. . . . . PERIOD . . . . . DATE . . . . . .
INSTRUCTOR'S COMMENTS . . . . . .

Experiment 17

Molecular Weight of a Gas

PURPOSE: To determine the molecular weight of a common gas experimentally.

APPARATUS: Balance, sensitive to 0.01 g; beaker, 250 ml; bottle, wide mouth; clamp, buret; drying tube; flask, Florence, 250 ml; funnel tube; glass bend, 8 × 8 cm; glass tube, 20 cm; glass tubing, 6 mm; graduated cylinder; ring stand; rubber connector; rubber tubing, connector; rubber stoppers, 1-hole No. 2, 1-hole No. 4, 2-hole No. 6; thermometer.

MATERIAL: Calcium carbonate, marble chips; calcium chloride, anhydrous, 4–8 mesh; cotton; hydrochloric acid, concentrated.

INTRODUCTION: You have learned that 1 mole of any gas occupies 22.4 liters at STP. Thus the mass, in grams, of 22.4 liters of a gas measured at standard conditions is numerically equal to the molecular weight of the gas. In this experiment, you will collect a container of a gas and determine its mass and volume under laboratory conditions. By application of the gas laws, you may determine the volume this mass of gas would occupy at STP. Then by simple arithmetic, the mass of 22.4 liters may be determined.

It is not practical to weigh an *empty* flask, that is, one completely devoid of gas molecules. You may, however, weigh a flask filled with air. Then, knowing the volume of the flask and the density of the air, the mass of air in the flask may be calculated. The difference in masses yields the mass of the *empty* flask.

PROCEDURE: 1. Place 20 g of calcium carbonate (marble chips) in a wide-mouth bottle and just cover with water. Assemble a 2-hole stopper, a glass bend, and a funnel tube to complete the generator. The end of the funnel tube should extend below the surface of the water in the wide-mouth bottle when the stopper assembly is in place. Why? Fill a drying tube with anhydrous calcium chloride, fitting a loose cotton plug in each end. Fit a 1-hole rubber stopper, in which is inserted a short length of glass tubing, into the open end of the drying tube. Clamp the assembled drying tube in a horizontal position on a ring stand and connect one end of the glass bend of the generator as shown in Fig. 17-1.

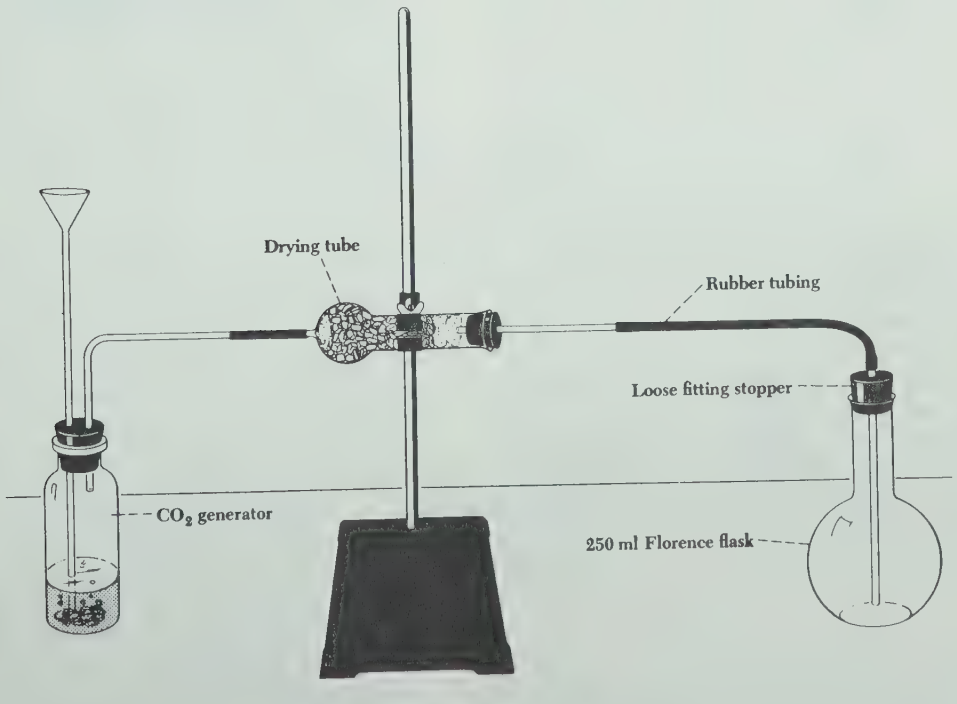

Fig. 17-1

2. Clean and thoroughly dry a 250-ml Florence flask. Select the largest 1-hole stopper which will just fit the flask and insert a glass tube about 20 cm in length so it extends to within 1 cm of the bottom of the flask. Determine the mass of the flask (which contains air at room temperature and pressure) with the stopper assembly in place to the nearest 0.01 g. *From this point on, handle the flask only by the rim.* Why? Record this mass in the Data Table. Place the flask on a sheet of clean paper (why?) and attach it to the drying tube by means of a suitable length of rubber tubing. *The stopper must be placed loosely in the neck of the flask.* It must not seal the flask. Why?

3. With the apparatus assembled as shown in Fig. 17-1 and the stopper loose on the Florence flask, you are ready to commence the generation of carbon dioxide. Add concentrated HCl a little at a time through the funnel tube to maintain a gentle evolution of carbon dioxide. Continue the delivery of CO_2 into the flask for about 20 minutes. You may *momentarily* tighten the stopper on the flask from time to time and observe that the liquid in the generator backs up in the funnel tube. How does this indicate that CO_2 is being delivered to the flask?

4. Detach the rubber hose from the flask, *being careful to handle the flask by the rim only*. Tighten the stopper assembly and weigh the stoppered flask of carbon dioxide gas. Record. Immediately remove the stopper and insert a thermometer directly into the flask. Record the temperature of the carbon dioxide. Record the laboratory barometer reading. Now fill the flask with water to the bottom of the stopper. Measure the volume of water contained by transferring it to a graduated cylinder. This corresponds to the volume of air first contained in the flask, and the volume of CO_2 collected.

CALCULATIONS: 1. *Mass of CO_2 collected.* The density of air may be taken as 1.29 g/liters at STP. To determine the mass of air contained in the flask, first correct the volume of air in the flask at the recorded temperature and pressure to STP. Then calculate the mass of this corrected volume of air. Knowing the mass of the flask containing air and the mass of the contained air, the mass of the "empty" flask may be computed and recorded. Determine the mass of CO_2 collected and record.

2. *Volume of CO_2 at STP.* The volume of CO_2 at STP is the same as the volume of air at STP calculated above. Record.

3. *Molecular weight of CO_2.* Knowing the mass of a given volume of CO_2 gas at STP, you may calculate the mass of the molar volume (22.4 liters at STP) of the gas. This mass in grams is numerically equal to the molecular weight of the CO_2 (experimental). Record.

4. *Experimental error.* Determine the correct molecular weight of carbon dioxide from the formula. The difference between your experimental value and this value represents your experimental error. Calculate your percentage error based on the correct molecular weight of the gas. Record your experimental and percentage error.

DATA TABLE

Mass of flask and stopper assembly, filled with air	g
Mass of flask and stopper assembly, filled with CO_2	g
Temperature of the CO_2	°C
Barometer reading	mm
Volume of the flask	ml
Mass of empty flask and stopper assembly	g
Mass of CO_2 collected	g
Volume of CO_2 at STP	ml
Molecular weight of CO_2 (experimental)	
Molecular weight of CO_2 (from molecular formula)	
Experimental error	
Percentage error	%

Experiment 18

NAME . . . . . PERIOD . . . . . DATE . . . . . .

INSTRUCTOR'S COMMENTS . . . . . .

Chemical Properties of Water

PURPOSE: To learn some of the chemical properties of water.

APPARATUS: Balance, platform; beaker, 150 ml; bottle, wide mouth; burner and tubing; clamp, test tube; dish, evaporating; flask, Erlenmeyer; forceps; 2 glass plates; graduated cylinder; spoon, deflagrating; test tube, Pyrex.

MATERIAL: Calcium, metal; calcium oxide, lump; copper(II) sulfate, crystals; phosphorus, red; potassium, metal; sodium aluminum sulfate, powdered; sodium hydrogen carbonate; sodium, metal; asbestos, paper; litmus paper, red and blue; solutions of lead(II) nitrate (0.1 *M*); potassium chromate (0.1 *M*).

INTRODUCTION: Water is a very stable substance that does not decompose easily. It does react, however, with a great many substances. Water is necessary for the formation of some crystals and is present in such crystals as water of hydration. Water promotes many chemical changes as is shown by the reaction which occurs when water is added to baking powder.

SUGGESTION: Use the lump calcium oxide from a newly opened bottle.

In place of sodium, use "dry-Na," containing approximately 8.8% sodium. This sodium-lead alloy (J. T. Baker Chemical Co.) is a convenient and moderately safe source of metallic sodium and is reasonably stable in air (*J. Chem. Educ.*, 48, 278 (1971).

PROCEDURE: 1. Obtain from your instructor a small piece of sodium metal, not larger than half a pea. Carry it to the laboratory desk of a dry glass plate. Always use forceps to handle sodium; never touch it with your fingers! Hold a glass plate in one hand to act as a shield between you and the reaction, and with your other hand drop the sodium into an evaporating dish that is half full of water. **CAUTION:** *Stand aside! Sometimes a slight explosion scatters particles of sodium which are caustic to the flesh.* Result? After the reaction is complete, dip a stirring rod into the liquid in the evaporating dish and touch it to a piece of red litmus paper on a clean glass plate. What happens? (Solutions of metallic hydroxides turn red litmus blue.)

Repeat the experiment using a small piece of potassium metal. CARE! How does this reaction compare in violence with that of sodium and water? What is the effect of the resulting solution on a strip of red litmus paper?

Drop a small piece of calcium metal into a test tube which is one quarter full of water. Result? Perform the flaming splint test for hydrogen. Dip a stirring rod into the liquid in the test tube and touch it to a piece of red litmus. Result?

Observations: Student answers. . . . . .

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2. Place a lump of calcium oxide about the size of a lima bean on a glass plate. Pour not more than 2 ml of water on the calcium oxide, until it becomes quite wet. It may take a minute or two for the reaction to occur. Result? After the reaction is over, transfer some of the residue on a stirring rod or spatula to a piece of moist red litmus paper on a clean glass plate.

Observations: Student answers. . . . . .

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3. Prepare the oxide of a nonmetal by igniting a piece of red phosphorus the size of a match head in the asbestos-lined bowl of a deflagrating spoon. Lower the spoon with the burning phosphorus into a wide-mouth bottle. Partly cover the bottle with a glass plate so as to retain as much of the smoke in the bottle as possible. Allow the phosphorus to burn out completely before removing the deflagrating spoon. Now add 30 ml of water to the bottle, and quickly cover the bottle tightly with the glass plate. Shake the bottle vigorously for a minute or two until the smoke disappears. What happens to it? Test the liquid in the bottle with a piece of blue litmus paper. Result? (Acid solutions turn blue litmus red.)

Observations: Student answers. . . . . .

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4. Place a crystal of copper(II) sulfate the size of a pea in a dry Pyrex test tube, and heat the test tube gently with a low burner

flame. Hold the test tube with the open end inclined slightly downward. As the reaction proceeds, you should be able to tell why this is necessary. What happens to the copper(II) sulfate crystal? Observe the substance which appears near the open end of the test tube. Identify this substance, give its source, and tell why it appeared near the open end of the test tube. After the test tube cools, add a drop of water to the residue in the test tube. Result. Why?

Observations: Student answers.

5. Mix 1 g of powdered sodium aluminum sulfate and 2 g of sodium hydrogen carbonate on a square of paper, and then add the mixture to a dry Erlenmeyer flask. Is there any evidence of chemical action? Pour 30 ml of water into the flask. What do you observe? Describe. (These substances are used in some types of baking powders.)

Observations: Student answers.

6. Add 5 ml of the lead(II) nitrate solution to 5 ml of the potassium chromate solution. Result? Consult the Table of Solubilities in the text and explain the result.

Observations: Student answers.

QUESTIONS: 1. What are the products of the reaction between the very active metals and water? Metallic hydroxide and hydrogen.

2. What class of compounds is produced when water reacts with the oxide of a metal? Metallic hydroxide.

3. What kind of compound is produced when water reacts with the oxide of a nonmetal? An acid.

4. Why do a definite number of molecules of water combine with a definite amount of a compound in forming a hydrate? Because a definite number of water molecules surround either or both ions of the compound in crystals of the hydrate.

5. Why is baking powder always stored in watertight containers? Because water causes the components of the baking powder to react with one another.

EQUATIONS: *Write balanced formula equations.*

1. Action of sodium on water.

$2Na + 2H_2O \rightarrow 2NaOH + H_2(g)$

2. Action of potassium on water.

$2K + 2H_2O \rightarrow 2KOH + H_2(g)$

3. Action of calcium on water.

$Ca + 2H_2O \rightarrow Ca(OH)_2 + H_2(g)$

4. Action of calcium oxide on water.

$CaO + H_2O \rightarrow Ca(OH)_2$

5. Action of diphosphorus pentoxide (P_4O_{10}) on water forming phosphoric acid (H_3PO_4).

$P_4O_{10} + 6H_2O \rightarrow 4H_3PO_4$

6. Action of heat on copper(II) sulfate crystals.

$CuSO_4 \cdot 5H_2O \rightarrow CuSO_4 + 5H_2O(g)$

7. (a) Reaction between solutions of $Pb(NO_3)_2$ and K_2CrO_4.

$Pb(NO_3)_2 + K_2CrO_4 \rightarrow PbCrO_4 + 2KNO_3$

(b) The net ionic equation for (a).

$Pb^{++}(aq) + CrO_4^{--}(aq) \rightarrow Pb^{++}CrO_4^{--}(s)$

Experiment 19

NAME . . . . . PERIOD . . . . . DATE . . . . . .

INSTRUCTOR'S COMMENTS . . . . . .

(a) Percentage of Water in a Hydrate

(b) Water of Crystallization

PURPOSE: (a) To find the percentage of water in crystallized barium chloride. (b) To determine the formula of the hydrate.

APPARATUS: Asbestos square, or bright wire gauze; balance, sensitive to 0.01 g; burner and tubing; crucible and cover; forceps; ring, iron; ring stand; tongs; triangle, pipestem.

MATERIAL: Barium chloride, C.P., crystals. Clear crystals of magnesium chloride, magnesium sulfate, potassium nitrate, sodium carbontae, sodium sulfate; crystals of copper(II) sulfate, potassium chloride, sodium aluminum sulfate, sodium chloride; calcium chloride, anhydrous, granular.

INTRODUCTION: Many ionic compounds, when crystallized from water solution, take up definite proportions of water as an integral part of their crystal structures. This water of crystallization may be driven off by the application of heat. Since the law of definite composition holds for crystalline hydrates, the number of moles of water of crystallization driven off per mole of the anhydrous compound is some simple number. If the formula of the anhydrous compound is known, you can then determine the formula of the hydrate.

PROCEDURE: 1. Arrange a suitable support for heating a crucible over a burner; use an asbestos square or preferably a clean, bright wire gauze for cooling the crucible. Throughout the experiment, handle the crucible and cover with clean crucible tongs only. Why?

Preheat a clean, dry crucible and cover to redness, cool, then determine their mass to the nearest 0.01 g. Add approximately 5 g of fine barium chloride crystals to the crucible and determine its mass to the nearest 0.01 g by the indirect method. Heat the crucible, contents, and cover gently at first (why?), increasing the temperature gradually, and finally heating strongly for 10 to 15 minutes. Cool and again determine the mass of the crucible, contents, and cover. (Keep a labeled record of each weighing as suggested in Experiment 1.)

2. Heat the covered crucible and contents again to redness for five minutes. When cool, reweigh. If the last two mass determinations differ by no more than 0.01 g, you may assume that the water has all been driven off. Otherwise repeat the process, heating to constant mass. The dehydrated compound left in the crucible should be returned to your instructor, since it can be used in the preparation of solutions.

Observations: Student answers. . . . . .

. . . . . .

CALCULATIONS: 1. *The percentage of water in crystallized barium chloride.* Knowing the mass of crystallized barium chloride used and the mass of the anhydrous compound which remained after heating, the mass of water of crystallization lost during the heating becomes evident. This loss of mass (the mass of water of crystallization) may be expressed as a percentage of the mass of the hydrates as follows:

$$\frac{\text{mass of water lost by heating}}{\text{mass of crystallized barium chloride}} \times 100\% =$$

$$\frac{\qquad\qquad}{\qquad\qquad} \times 100\% = \qquad\%$$

This is your *experimental* result. Now calculate the theoretical percentage of water of crystallization in crystallized barium chloride from the correct formula, $BaCl_2 \cdot 2H_2O$. Show your computations below in smooth form on the back of this page and enter the results in the Data Table.

DATA TABLE

Mass of empty crucible and cover . .	g
Mass of crucible, cover and crystallized barium chloride	g
Mass of crystallized barium chloride	g
Mass of crucible, cover, and anhydrous barium chloride	g
Mass of anhydrous barium chloride.	g
Mass of water lost by heating	g

Percentage of water in crystallized barium chloride . . . . . %
Theoretical percentage of water in crystallized barium chloride . . %
Experimental error . . . . . %
Percentage error . . . . . %

2. *The formula of crystallized barium chloride.* The *g-formula wt.* of crystallized barium chloride is in the same ratio to the *g-formula wt.* of anhydrous barium chloride as the *actual mass* of crystallized barium chloride used is to the *actual mass* of anhydrous barium chloride recovered. You have the masses of these two substances, and you can compute the g-formula wt. of anhydrous $BaCl_2$ from the atomic weights.

g-formual wt. of crystallized barium chloride . . . . . g

g-formula wt. of anhydrous barium chloride . . . . . g

part of the g-formula wt. of the hydrate due to water . . . . . g

number of moles of water per g-formula wt. of hydrate . . . . . moles
(calculate to the nearest whole number)

formula of crystallized barium chloride

. . . . . .

The g-formula wt. of crystallized barium chloride can then be found by means of the following proportion:

$$\frac{\text{g-formula wt. of cryst. barium chloride}}{\text{g-formula wt. of anhyd. barium chloride}} = \frac{\text{g of cryst. barium chloride used}}{\text{g of anhyd. barium chloride left}}$$

3. In *dry* test tubes, heat successively *one* or *two* small crystals of as many of the compounds listed in the table below as your instructor may direct. In most of the cases only moderate heating is required. Note the effects. Tabulate the results.

QUESTION: How does the percentage of water in crystallized barium chloride exemplify the law of definite composition? . . . .

Every compound has a definite composition by weight. . . . . Pure crystallized barium chloride always has the same composition. . . . . .

FURTHER EXPERIMENTATION: In the table below investigate the specific properties of several familiar crystalline hydrates to determine others which lend themselves to this method of determining percentage hydration.

Collect data on some representative substances and verify their hydrate formulas.

Undertake an explanation of any large deviations from the correct hydrate formulas that your data may yield.

Name of compound	Water of crystallization present?	Description of residue
sodium carbonate	yes	white powder
sodium sulfate	yes	white powder
sodium chloride	no	unchanged
copper(II) sulfate	anhydrous (no)	unchanged
sodium aluminum sulfate	yes	white powder
potassium chloride	no	unchnaged
magnesium chloride	no	unchanged
potassium nitrate	no	may melt

NAME. . . . . PERIOD . . . . . DATE . . . . . .
INSTRUCTOR'S COMMENTS . . . . . .

Experiment 20 Solution and Molecular Polarity

PURPOSE: To show how the nature of the attractive forces between molecules determines and helps to explain the solution process and the selectivity of solvents.

APPARATUS: Graduated cylinder (10 ml); microspatula (or wooden splint); stoppers, cork; test tubes, Pyrex, small.

ADDITIONAL APPARATUS FOR INSTRUCTOR'S EXPERIMENT: 2 bottles, wide-mouth, drying tube; hydrogen chloride generator.

MATERIAL: Iodine, solid; benzene; carbon tetrachloride; ethanol; ethylene glycol; glycerol.

ADDITIONAL MATERIAL FOR INSTRUCTOR'S EXPERIMENT: calcium chloride; sodium chloride; litmus papers, red and blue; sulfuric acid, concentrated.

SUGGESTION: If students are not permitted to use carbon tetrachloride, tetrachloroethylene (C_2Cl_4, perchloroethylene) may be substituted.

INTRODUCTION: A rough rule which applies to the phenomenon of solubility is: "like dissolves like." In general, the possibility of solution taking place is increased by a similarity in the composition and structure of the substances involved. The forces between molecules which play a role in the solution process include (1) the dispersion interaction forces which are common to all atoms and molecules, both polar and nonpolar, (2) the dipole forces between polar molecules, and (3) the forces related to hydrogen bonding. Consult Sec. 9.4, page 166 for dispersion interaction and Sec. 11.16, page 215 for hydrogen bond in *Modern Chemistry*, 1974. Nonpolar substances dissolve in nonpolar liquids because of dispersion interaction. Polar liquids and substances dissolve in polar liquids because of dipole-dipole interaction and dispersion interactions. Hydrogen bonding may also play a role in the solution of polar substances. Some nonpolar substances do dissolve in polar liquids because of the magnitude of the dispersion interactions. For a particular substance, these three types of forces play an important role in determining its melting point, boiling point (condensation temperature), vapor pressure, and critical temperature. The hydrogen bonding in water is responsible for its unusually high melting and boiling points. Hydrogen bonds also exist in alcohols such as ethanol (C_2H_5OH), ethylene glycol ($C_2H_4(OH)_2$), and glycerol ($C_3H_5(OH)_3$). The molecules of these water-related compounds are also dipoles.

One measure of the polarity of a substance is the measurable property known as the dielectric constant. (You may wish to refer to a physics textbook for a detailed explanation.) The relative magnitudes of the dielectric constants of the liquids used in this experiment are listed in the following table.

Substance	*Dielectric constant (20° C)*
carbon tetrachloride, CCl_4	2.238
benzene, C_6H_6	2.284
hydrogen chloride, HCl (−15° C)	6.35
ethanol, C_2H_5OH	24.30
ethylene glycol, $C_2H_4(OH)_2$	37.
glycerol, $C_3H_5(OH)_3$	42.5
water, H_2O	80.37

As part of this experiment, you will test the solubility of one nonpolar solid, iodine, which exists as a molecular crystal composed of I_2 molecules. The Instructor experiment deals with the solubility of hydrogen chloride, a polar substance.

SUGGESTIONS: Students should measure out the recommended volume of water, generally 5 ml, in the 10 ml graduated cylinder. They should approximate this measured volume by using similar small size test tubes and a dropper. To prevent waste of materials and promote safety, the liquids should be dispensed from stoppered bottles having long-stemmed droppers through the stopper.

CAUTION: During this experiment the following directions *must* be followed because some of the substances used are highly toxic or combustible. All laboratory burners are to be extinguished. Benzene, carbon tetrachloride, and ethylene glycol are *poisonous*. Benzene is *combustible*. Therefore all liquids are to be used in 1 ml quantities. Shaking of mixtures is to be performed only in cork-stoppered test tubes. Iodine must be handled with a microspatula so that the

DATA TABLE 1

I_2 in	Soluble or insoluble	Color of solution
water	practically insoluble	may have very faint brown
ethanol	soluble	brown
benzene	soluble	brown
carbon tetrachloride	soluble	violet

recommended quantity of two tiny crystals is used in each case.

PROCEDURE: 1. *Solubility and the polarity of solvents.* Measure out 5 ml of water with the graduated cylinder and pour into the test tube in the test tube rack. Place 5 ml of ethanol, benzene, and carbon tetrachloride into successive test tubes approximating the volume of water in the first test tube. Add 2 small iodine crystals to each. Stopper and shake vigorously. Report evidence of solubility and the resultant color of solutions, when produced, in Data Table 1.

2. *Comparative polarity of liquids.* Into each of four separate test tubes add 5 ml of benzene. To the first test tube add 5 ml of water, to the second 5 ml of ethanol, to the third 5 ml of ethylene glycol, and to the fourth 5 ml of glycerol. Stopper and shake each test tube. Report the existence of one layer which indicates miscibility or that of two separate layers indicating nonmiscibility. Save the test tube contents for Part 3.

Observations: The alcohol and benzene are completely miscible. The 3 other samples are immiscible, having two layers.

Basing your judgment on the results of Part 2, how would you expect a mixture of benzene and carbon tetrachloride to behave when shaken? Make this test, using 1 ml of each liquid, and record its result.

Observations: Since CCl_4 and benzene are both nonpolar they dissolve in each other, they are miscible. Experimental results confirm this inference.

3. Add 5 ml of ethanol to each of the test tubes, saved from Part 2, except the one containing ethanol. Stopper and shake vigorously. Look for the existence of more than one layer and the height of the lower layer compared to that of the upper layer. Where benzene is immiscible, it will float on top of the other liquid(s). Record your results in Data Table 2.

On the basis of the results obtained, list the liquids; glycerol, ethanol, and ethylene glycol in order of increasing polarity:

a. ethanol *b.* ethylene glycol *c.* glycerol

4. INSTRUCTOR EXPERIMENT: Place the end of the delivery tube from the hydrogen chloride generator one-quarter of an inch above the surface of a one-inch layer of water in a gas collecting bottle. Set the generator into operation. Students should note the appearance of apparent convection currents of an oily substance where the hydrogen chloride comes into contact with the water. What does this indicate about the solubility of HCl in water? Add blue litmus to the contents of the bottle. Students should note results and explain evidence of solubility of hydrogen chloride in water. Now permit dry hydrogen chloride [dry by passing through $CaCl_2$ or H_2SO_4

DATA TABLE 2

Addition of ethanol to:	No. of layers at start	No. of layers at finish	Height of lower layer
benzene + water	2	2	about 2/3
benzene + ethylene glycol	2	2	about 19/20
benzene + glycerol	2	2	about 2/3

(conc.)] to pass into and through one inch of benzene in a small gas collecting bottle. Have residual hydrogen chloride gas blown out of the top of the bottle with air. Add litmus (blue) to benzene. Now add one inch of water to the bottle, shake vigorously and inspect the litmus paper in the water layer.

What is its color? Red

Where did the hydrogen chloride come from? HCl was only slightly soluble in the benzene. It dissolved in the water.

What does this result indicate about the comparative solubility of hydrogen chloride in water as compared with its solubility in benzene? What does this indicate about polarity of HCl? HCl is much more soluble in water since HCl is polar.

The conductivity apparatus (page 246 in MODERN CHEMISTRY, 1974) may be used to test for electrical conductivity of 1. HCl in benzene, and 2. HCl in benzene + water.

QUESTIONS:

1. Explain why iodine is soluble in benzene and carbon tetrachloride. Iodine crystals are composed of nonpolar molecules. Benzene and carbon tetrachloride are nonpolar liquids having large molecular weights (and large number of electrons) and hence relatively large van der Waals forces.

2. Explain why iodine dissolves in ethanol but practically not at all in water. Although ethanol is slightly polar, its van der Waals forces are large enough to cause the solution of iodine. Water is very polar and the magnitude of its van der Waals forces is less than that of ethanol.

3. Look up and record the dielectric constant of iodine: Solid: 4; Liquid (118°C: 11.

4. Ethanol has the formula, C_2H_5OH, ethylene glycol, $C_2H_4(OH)_2$, and glycerol $C_3H_5(OH)_3$. What apparent effect does increasing the number of hydroxyl groups in a compound have on its polarity? Increasing the number of hydroxyl groups increases the polarity of the molecule.

5. Explain the difference in results which occurred when ethanol was shaken with (1) benzene and ethylene glycol and (2) benzene and glycerol. Since practically no benzene dissolved in the mixed lower layer of ethanol + glycerine while some benzene dissolved in the lower layer of ethanol + ethylene glycol, glycerol is more polar than ethylene glycol. All the polar liquids are miscible because of polarity and possibly hydrogen bonding.

6. Cite experimental evidence which indicates that nonpolar liquids (substances) are soluble in polar liquids having a wide range in polarity. Benzene is soluble in ethanol whose dielectric constant is 24.30 indicating considerably polarity. Furthermore, benzene dissolved to some extent in the mixture of ethanol and ethylene glycol (average of dielectric constants approximately 31). Note: Have students try the solubility of benzene in CS_2 and methanol. Have them look up the dielectric constants of these substances and relate to the answer to question 7.

7. Cite experimental evidence that a nonpolar liquid may be insoluble with liquids of high polarity. Benzene is not soluble in ethylene glycol, glycerol, or water.

8. Cite experimental evidence which indicates that "like dissolves like" as it relates to preference of polar liquids for each other rather than for nonpolar liquids. The relative heights of the mixtures in Part 3 indicated that the polar liquids dissolved in each other rather than with benzene (nonpolar), the effect being most marked with water and with glycerol.

Experiment 21

NAME. . . . . PERIOD . . . . . DATE . . . . . .
INSTRUCTOR'S COMMENTS . . . . . .

The Solid State, Crystals and Crystallization

PURPOSE: To show various processes by which crystals are formed and to study the nature of crystals.

APPARATUS: Balance, platform; beaker; burner and tubing; clamp, test tube; dish, evaporating; funnel; glass plate; graduated cylinder; hand lens; ring, iron; ring stand, spatula; thermometer; watch glass; wire gauze.

ADDITIONAL APPARATUS FOR INSTRUCTOR'S EXPERIMENT: Graduated cylinder; test tube; watch glass.

MATERIAL: Ammonium chloride; ice; iodine; sodium chloride; sulfur, powder; filter paper.

ADDITIONAL MATERIAL FOR INSTRUCTOR'S EXPERIMENT: Sulfur, powder; carbon disulfide.

SUGGESTION: Amorphous solids: To demonstrate or have your students prepare amorphous sulfur refer to Exp. 58, Forms of Sulfur, Procedure 3. An alternate procedure is to have the students heat a piece of glass tubing or a glass rod in the burner flame and permit it to bend under its own weight as the solid softens.

INTRODUCTION: The properties characteristic of the solid state are evident from the study of true solids composed of crystals. In crystalline solids, the particles have a regular arrangement resulting in a definite geometric shape. A specific crystal has a definite number of faces so arranged that the interfacial angles between adjacent faces are definite. The classes of crystals, according to their shape, are isometric (cubic), tetragonal, triclinic, hexagonal, orthorhombic, and monoclinic. The definite shape, definite volume, and incompressibility of solids is due to the forces which hold the particles together in fixed positions. Crystalline solids have definite melting points whose magnitudes indicate the nature of the binding forces which hold the particles together in the crystal lattice. Crystals may be classified according to the types of lattice structure, that is, whether the particles that compose the crystal lattice are ionic, covalent, metallic, or molecular. Perfectly shaped crystals rarely occur in nature. Examination of a laboratory specimen of quartz generally reveals a jumble of imperfectly shaped crystals joined together. The formation of multiple crystals tends to interfere with the development of single perfect crystals. In this experiment, the various processes by which natural forces arrange the particles of substances into crystals will be studied. These are cooling a molten mass, evaporation from solution, cooling a hot saturated solution, and sublimation.

PROCEDURE: 1. *Crystallization. a.* Put 5 grams of powdered roll sulfur into a test tube. Holding the test tube in a clamp, gently heat the contents. Note the color of the sulfur as it melts in contact with the hot glass. Continue to heat gently until all the sulfur liquefies. Avoid over-heating and try to keep the color of the final melt close to that of the initially melted sulfur. If the sulfur darkens due to local over-heating, stop heating for a short time and gently shake the tube contents (shake in a nearly horizontal, not vertical plane). Pour the liquid sulfur into a folded dry filter paper placed in a funnel. When a crust forms over the fold, remove the paper and quickly open the fold. Permit any excess liquid sulfur to run onto a piece of paper rather than onto the table top. Describe the appearance of the crystals that have been formed and state the crystal system to which they belong.

Observations: Needle-shaped crystals, monoclinic. . . . . .

. . . . . .

b. Put 3 or 4 small pieces of solid iodine in an evaporating dish using a spatula or wooden splint. Place the dish on the wire gauze and cover the dish with a watch glass (convex side down). Pour 10 ml of water into the watch glass. Heat the evaporating dish with a low flame. See Fig. 21-1. Note the color of the iodine vapor. Continue to heat until most of the solid has vaporized. Permit the apparatus to cool. Remove the watch glass, discard the water, and examine the deposit on the bottom of the watch glass. Note the color and shape of the solid crystals. Scrape off a small portion of the deposit onto a glass plate. Examine under the hand lens. You may be able to detect a crystal which has the characteristic shape of solid iodine. To which crystal system does it belong? Wash off the iodine deposit with hot water. **CAUTION:** *Avoid contact of solid iodine with the skin.*

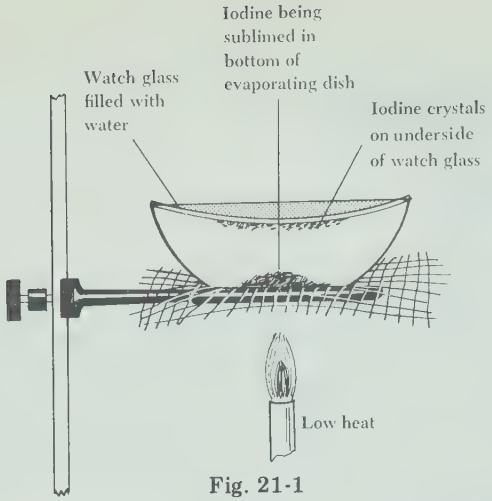

Iodine being sublimed in bottom of evaporating dish
Watch glass filled with water
Iodine crystals on underside of watch glass
Low heat

Fig. 21-1

Observations: I_2 vapor is violet. The violet-black crystals with a metallic luster form as needles. Have students consult handbook (I_2 crystals are rhombic).

c. Into 10 ml of water in a test tube pour 5 grams of ammonium chloride. Shake thoroughly. To what extent does it dissolve? If necessary, heat gently until all the ammonium chloride dissolves. Add another 5 grams of ammonium chloride and continue to heat with shaking until it all dissolves. Now permit cold tap water to run over the outside of the test tube until you note signs of crystallization. Note the appearance of the crystals. Permit the crystals to settle out. Remove some of them with a splint. Place the crystals on a piece of filter paper to remove most of the water. Transfer a portion of the sample to a glass plate and examine under the hand lens. Describe the shape and size of the crystals you see.

Observations: The small crystals appear almost as a "snow." Examination under the hand lens may result in students identifying as belonging to cubic system.

d. INSTRUCTOR EXPERIMENT. Use procedure 1 of Exp. 58, Forms of Sulfur, for preparation of rhombic sulfur. Add 2 grams of powdered roll sulfur to 5 ml of carbon disulfide in a test tube. **CAUTION:** *All open flames should be extinguished since the vapors of carbon disulfide are dangerously combustible.* Have students note any evidence of solution, i.e., color of solution. Permit the excess solid sulfur to settle, and pour the clear solution into a watch glass. Place the watch glass under the hood and partially cover with a beaker, one side of which is supported by a match box. Students should explain the effect of this arrangement on the rate of carbon disulfide evaporation and hence the rate of crystal formation. When evaporation is complete, students should examine the watch glass deposit and samples from it under the hand lens. Students should describe and identify the crystal shape, and compare the size of these crystals (produced slowly) with those of ammonium chloride (produced rapidly). The teacher may wish to use the overhead projector in this demonstration or an alternate using a drop of sodium chloride solution on a microscope slide.

2. *Crystal Properties. a.* Examine several grains of sodium chloride under the hand lens. Note the shape of a single crystal. How does the shape of this crystal compare with that of the other crystals that you can also see? You have previously examined two different samples of sulfur crystals. Name the crystal system to which the sodium chloride crystals belong.

Observations: NaCl are cubic crystals.

b. Put several cubes or chunks of ice into a small beaker and insert a thermometer. Permit the ice to melt and record three readings of the thermometer at 5 minute intervals during the period (some ice must always be present). What property of a crystal is revealed by this procedure?

Observations: The melting point of a true solid is a constant property.

QUESTIONS: 1. State two characteristics of crystals. Crystals are bounded by plane surfaces making definite angles with each other, giving a regular geometric form.

2. Name three crystalline systems of which you observed at least one example in this experiment. Isometric or cubic, orthorhombic, monoclinic.

3. Name or briefly describe the four different crystallization processes. Cooling a molten mass, evaporation from solution, cooling a hot concentrated solution until saturation is reached, and sublimation.

4. *a.* To what state does a solid usually change upon being heated?

b. To what state does a solid go when it undergoes sublimation?

a. Liquid.

b. Vapor.

5. Explain the apparent "disappearance" of such solids as Dry Ice and naphthalene moth balls (crystals). The solid vaporizes directly without passing through the liquid phase because it has an equilibrium vapor pressure which rises very rapidly with increase in temperature.

6. What condition determines the size of crystals being produced by crystallization processes? The speed of crystallization, slower crystallization resulting in larger crystals.

Experiment 22

NAME . . . . . PERIOD . . . . . DATE . . . . . .
INSTRUCTOR'S COMMENTS . . . . . .

Solubility, Rate of Solution, Heat of Solution

PURPOSE: To study some of the factors affecting the solubility and the rate of solution of a solid in water. To observe evidence that the heat of solution of a substance is related to the effect of temperature on its solubility.

APPARATUS: Balance, platform; beaker, 400 ml; burner and tubing; clamp, test tube; 2 flasks, Erlenmeyer, 125 ml; graduated cylinder; mortar and pestle; rubber stoppers, solid, No. 2; spatula; test tubes; thermometer.

ADDITIONAL APPARATUS FOR INSTRUCTOR'S EXPERIMENT: Beaker, 100 ml; burner and tubing; dish, evaporating, large; funnel, large; ring, iron; ring stand; thermometer; wire gauze.

MATERIAL: Ammonium chloride; potassium nitrate; sodium hydroxide (pellets).

ADDITIONAL MATERIAL FOR INSTRUCTOR'S EXPERIMENT: Filter paper (large); sulfuric acid, concentrated.

INTRODUCTION: The three factors that hasten the solution process of a solid in water will be studied in this experiment. You will observe the effect of temperature changes on the quantity of solid that can be dissolved in a given amount of water. You will also observe that some solids dissolve in water with the absorption of heat while others dissolve with the evolution of heat. This indicates that the heat of solution of a particular substance plays a role in the effect of temperature changes on the solubility of the substance. Solutes with positive heats of solution become more soluble as the temperature of their solution is raised. The effect of temperature on saturated solutions of solutes having negative heats of solution is the reverse.

SUGGESTION: Two students may work together, thereby reducing the quantity of KNO_3 solution to be processed by the instructor. A clean container of adequate volume shuold be designated in which to collect the KNO_3 solutions prepared by the students in Parts 2, 3, 4, and 5. The instructor may wish to delegate the task of recovering the KNO_3 in crystalline form to one or two interested students as an extra project.

PROCEDURE: 1. *a.* Put 10 ml of water in each of three test tubes. Insert a thermometer into the water in the first test tube and record the temperature. To the water in the first test tube, add 5 g of potassium nitrate. Gently stir the tube contents with the stirring rod. Note the temperature when all 5 g have been dissolved, or almost completely dissolved. Repeat this procedure with the second test tube using 5 g of ammonium chloride. To the third test tube add 5 pellets of sodium hydroxide, transferring them from the bottle to the test tube by using a spatula. **CAUTION:** *Sodium hydroxide is a powerful caustic hydroxide. Do not handle the pellets or permit any of the solution to touch your skin.* Record your results in the Data Table. Refer to the Heats of Solution Table in Chapter 12 of MODERN CHEMISTRY, 1974, or to a Handbook.

DATA TABLE

Solute	Water temperature °C		Heat of solution of solute (kcal/mole)
	Initial	Final (solution)	
KNO_3	. . . . .	. . . . .	positive (+ 8.52)
NH_4Cl	. . . . .	. . . . .	positive (+ 3.88)
NaOH	. . . . .	. . . . .	negative (−9.94)
H_2SO_4	. . . . .	. . . . .	negative (−17.75)

b. INSTRUCTOR'S EXPERIMENT. To 20 ml of water in a beaker add with stirring 5 ml of concentrated sulfuric acid. Have a student read the initial and final temperatures. Students should record this information in the Data Table.

2. Weigh out two 2-g portions of coarse crystals of potassium nitrate. Grind one portion to a fine powder, using a rotary motion of the pestle; *do not pound the crystals*. Have at hand two Erlenmeyer flasks each containing 25 ml of distilled water at room temperature. Add the two portions to the two flasks simultaneously and agitate similarly with swirling motions. Observe the time required for complete solution in each case. Explain the difference in time on the basis of surface area of solute. *Label a clean 400-ml beaker and pour all solutions prepared in Parts 2, 3, 4, and 5 into the beaker.*

Observations: The finely powdered KNO_3 dissolves much more rapidly because of the larger surface area which contacts the solvent.

3. Repeat the experiment, but pulverize both portions. Swirl one portion as before, but let the other stand without stirring. Compare the rates at which solution occurs. *Pour the solutions into the labeled beaker.*

Observations: The stirred, pulverized KNO_3 dissolves much more rapidly.

4. Repeat, using two 2-g portions of large crystals. Compare the rate of solution in one case using 25 ml of distilled water at room temperature, and in the other case 25 ml of distilled water heated to near the boiling point. Stir both solutions similarly. Pour the solutions into the labeled beaker.

Observations: The KNO_3 in the hot water dissolves much more rapidly.

5. Grind 5 g of potassium nitrate to a powder. **CAUTION.** To 10 ml of distilled water (at room temperature) in a test tube, add the powdered KNO_3 gradually, a little at a time. Stopper the test tube and shake it after each addition of solute. After all the KNO_3 has been added and the solution is saturated at room temperature (how do you know it is saturated?), warm *gently* over a low burner flame. *The stopper must be removed.* Move the test tube in and out of the flame carefully to avoid sudden, violent boiling. When the liquid appears to be near the boiling point, remove the test tube from the flame and examine the solution. Has all the KNO_3 dissolved at the higher temperature? Is the solution *saturated* or *unsaturated*? Justify your decision. Drop in a small crystal of KNO_3 to see if it dissolves. If not, warm the solution carefully again until all of the potassium nitrate is in solution. What do you conclude about the solubility of potassium nitrate in hot water, as compared with its solubility in cold water? (Refer to the solubility curves in Chapter 12 of MODERN CHEMISTRY, 1974.) Now allow the solution to cool. Devise a means of hastening the cooling process. Result? Reheat the solution to dissolve the crystals which formed and transfer all accumulated KNO_3 solution to a container designated by the instructor. Carefully summarize your observations concerning the influence of the temperature of the water on the solubility of the potassium nitrate.

Observations: No more solute will dissolve indicating a saturated solution at room temperature. All of the KNO_3 dissolves at the higher temperature since no more solid solute is evident. KNO_3 is much more soluble at higher and higher temperatures (in hot water). Upon cooling the hot solution, in running cold water, the KNO_3 starts to settle out to form a saturated solution at the lower temperature.

6. INSTRUCTOR'S EXPERIMENT. Filter the solutions of potassium nitrate collected from the students into a large evaporating dish. Evaporate to approximately one-half volume and, when cool, pour off the mother liquor and dry the crystals between sheets of filter paper. The process may be repeated, and a second or even a third crop of crystals may be obtained. When dry, return the crystals to a properly labeled stock bottle for future use.

QUESTIONS: 1. Explain why pulverizing a solid solute increases the rate of solution. Since solution action occurs only on the surface of the solid, grinding a solid to a powder increases total surface area and hence the rate of solution.

2. Why does stirring increase the rate at which most solids dissolve? Stirring or shaking brings fresh portions of the solvent in contact with undissolved solute.

3. Why does increasing the temperature, other conditions being kept constant, increase the rate of solution of potassium

nitrate? The molecular activity of the solvent increases and the solubility of KNO_3 is increased since the dissolving process is endothermic (KNO_3 has a positive heat of solution).

4. What effect would you expect increased temperature to have on the solubility of ammonium chloride? Why? The solubility of the ammonium chloride is increased for the same reasons as given in the answer to Q. 3 (above).

5. The heat of solution of lithium carbonate is −3.06 kcal/mole solute in 200 moles of H_2O. What effect would you expect increased temperature to have on the concentration of Li_2CO_3 in its saturated solution? Explain. It would decrease the concentration of the saturated solution since the heat of solution is negative.

FURTHER EXPERIMENTATION: Two other chemicals which may be used in experiments on the heat of solution are ammonium nitrate and calcium chloride. These are used in instant cold and hot packs as first aids devices. Consult the article, "Thermochemistry and First Aid," by Smith, W. L. *Chemistry*, 45, 18 (1972 — Sept.).

Experiment 23

NAME. . . . . PERIOD . . . . . DATE . . . . . .

INSTRUCTOR'S COMMENTS . . . . . .

A Solubility Curve

PURPOSE: To show how the solubility of a salt such as ammonium chloride varies with the temperature and how to plot its solubility curve on the basis of observed data.

APPARATUS: Balance, sensitive to 0.01 g; beaker, 400 ml; burner and tubing; pipet, 10 ml; ring, iron; ring stand; test tubes, Pyrex; thermometer; wire gauze.

MATERIAL: Ammonium chloride; graph paper.

INTRODUCTION: The solubility of a solute is the amount of solute dissolved in a given amount of a certain solvent at equilibrium, under specified conditions. The usual units used on solubility graphs are grams of solute per 100 ml of water at specified Celsius temperatures. Increasing the temperature usually increases the solubility of solids in liquids. Decreasing the temperature usually decreases the solubility. In this experiment we will determine the solubility of ammonium chloride at various temperatures by allowing a hot solution of the compound to cool and observing the temperature at which the solid begins to crystallize.

PROCEDURE: 1. Weigh out 4.00-g, 5.00-g, 6.00-g, and 7.00-g samples of ammonium chloride. Put each sample in a properly labeled test tube. Using a pipette, measure 10.0 ml of water into each test tube. Fill the beaker half full of water, put the test tubes containing the 4.00-g and 5.00-g samples in the water, and bring the water to a boil. Meanwhile, heat the test tube containing the 7.00-g sample gently with the burner, stirring continuously until all the solute dissolves. Do not bring the test tube contents to a boil. With the thermometer in the solution, allow the contents to cool while stirring gently. Record the temperature at which the solid beings to crystallize (when the first crystals appear). Heat the solution in the test tube again to dissolve the crystals, allow it to cool, and recheck the temperature at which crystallization occurs. Repeat this procedure with the test tube containing the 6.00-g sample. Repeat with the 5.00-g and 4.00-g samples using the hot water bath as a source of heat, rather than the burner. A cold water bath is needed to hasten the crystallization of the 4.00-g sample.

2. Plot the solubility curve for ammonium chloride from the results listed in your Data Table. Along the horizontal axis on the graph paper, write the temperature scale 0°C to 100°C using 10 degree intervals. Along the vertical axis, write the solubility in g/10 ml H_2O from 0 to 10.00 g intervals. Place dots corresponding to the four masses you used at the places corresponding to the observed temperatures. Draw a small circle around each of these points and construct a smooth curve for the observed data. Extrapolate this curve to the temperature limits by using a dashed (----) line to indicate extrapolation.

DATA TABLE

Solubility (g/10.0 ml H_2O)	Temperature
4.00 g	°C
5.00 g	°C
6.00 g	°C
7.00 g	°C

QUESTIONS: 1. From your solubility curve determine the solubility of ammonium chloride in g/100 ml H_2O at 60°C.

Student answer. . . . . .

2. Consult a Handbook and record the accepted value for the solubility of ammonium chloride at 60°C. . . . 55.2 g. . . . . .

. . . . . .

3. Calculate your percentage error from the answers to Questions 1 and 2. Percentage error. . . . Student answer. . . . . %

FURTHER EXPERIMENTATION: Repeat the procedure using other compounds. For $NaNO_3$ try samples starting with 8.00 g and going up by successive increments to 14.00 g. For KNO_3, try samples starting with 4.00 g and going up by increments of 3.00 g to 13.00 g. Evaluate the changes in solubilities of NH_4Cl, $NaNO_3$, and KNO_3. Construct solubility curves using molalities instead of grams per 100 ml of water.

Solubility Data for NH_4Cl in grams per 100 ml H_2O

30°C	41.4 g
50°C	50.4 g
70°C	60.2 g
90°C	71.3 g

Experiment 24

NAME. . . . . PERIOD . . . . . DATE . . . . . .

INSTRUCTOR'S COMMENTS . . . . . .

(a) Preparation and Properties of Acids

PURPOSE: To prepare some acids from their anhydrides. To study some common properties of acids.

APPARATUS: Balance, platform; beaker, 400 ml; 3 bottles, wide-mouth; burner and tubing; clamp, buret; clamp, test tube; flask, Florence; funnel, separatory; 4 glass plates; graduated cylinder; medicine dropper; ring stand; rubber stoppers, 1-hole No. 4, 2-hole No. 4; spoon, deflagrating; test tubes; 2 test tubes, hand glass; trough.

MATERIAL: Charcoal, wood, lumps; copper wire, 22-30 gauge; phosphorus, red; sodium peroxide; sulfur, roll; zinc, sheet, 1 cm^2 pieces; asbestos paper; litmus papers, red and blue; sandpaper; nitric acid, concentrated; acetic acid, dilute (1:4); hydrochloric acid, (1:3); phosphoric acid, dilute (1:14); sulfuric acid, dilute (1:11); solutions of methyl orange, phenolphthalein.

SUGGESTION: If a separatory funnel is not available, a suitable dropping funnel may be improvised from a short-stemmed funnel, iron ring, pinch clamp, rubber connector, and glass tubing.

Satisfactory results may be obtained by burning the substances in bottles of air if time does not permit the use of the oxygen generator. A small candle in a deflagrating spoon may be substituted for charcoal in the production of carbon dioxide in the bottle of air.

INTRODUCTION: Except for fluorine, oxygen is the most highly electronegative element. We would naturally expect many elements, both metals and nonmetals, to combine readily with oxygen. Some of the chemical properties of the oxides, notably their reactions with water, enable us to recognize the correlation between the elements and their positions in the Periodic Table. In general, oxides in reaction with water form oxygen-hydrogen groups. The character of the product may depend on the character of the associated element. Elements near the central region of the Periodic Table may show amphiprotic properties. Thus $Zn(OH)_2$ is the proper formula for zinc hydroxide in the presence of a strong acid. However, H_2ZnO_2 is more suitable when the same compound is in the presence of a strong hydroxide. In a general sense, oxides of the highly electropositive elements react with water to form solutions which are distinctly basic. These elements are located to the left of the central region of the Periodic Table. Oxides of the highly electronegative elements react with water to form solutions which are distinctly acidic. These elements are located to the right of the central region of the Periodic Table. Such nonmetallic oxides are known as acid anhydrides. In this experiment, you will prepare some typical acid anhydrides and observe some of the common properties of their water solutions.

It is true, of course, that some acids do not contain oxygen. Binary acids, such as hydrochloric and hydrosulfuric, will be studied in later experiments.

PROCEDURE: 1. Assemble an oxygen generator as shown in Fig. 24-1. The flask (Pyrex) to be used as the generator must be clean and *thoroughly* dry. **CAUTION:** *Sodium peroxide is a vigorous oxidizing agent and reacts with a limited supply of water to produce oxygen, releasing much heat in the process.* Weigh out 10 g of Na_2O_2 on a dry weighing paper and transfer it to the *dry* generator flask. Wet the weighing paper thoroughly with water before discarding. Why? Prepare three wide-mouth bottles to collect the O_2 by water displacement. When ready, ask your instructor to inspect the apparatus.

Close the stopcock of the funnel and partially fill it with water. Place a collecting bottle in position and open the stopcock just enough to allow water to enter the generator dropwise. Regulate the addition of water to provide a moderate evolution of oxygen. Discard the first bottle of oxygen collected. Why? Refill the bottle with water and return it to the trough. As the water in each bottle is displaced, cover the mouth with a glass square and place it mouth upward on the desk (covered). Feel the sides of the generator. Result? How would you describe the reaction in the generator? Con-

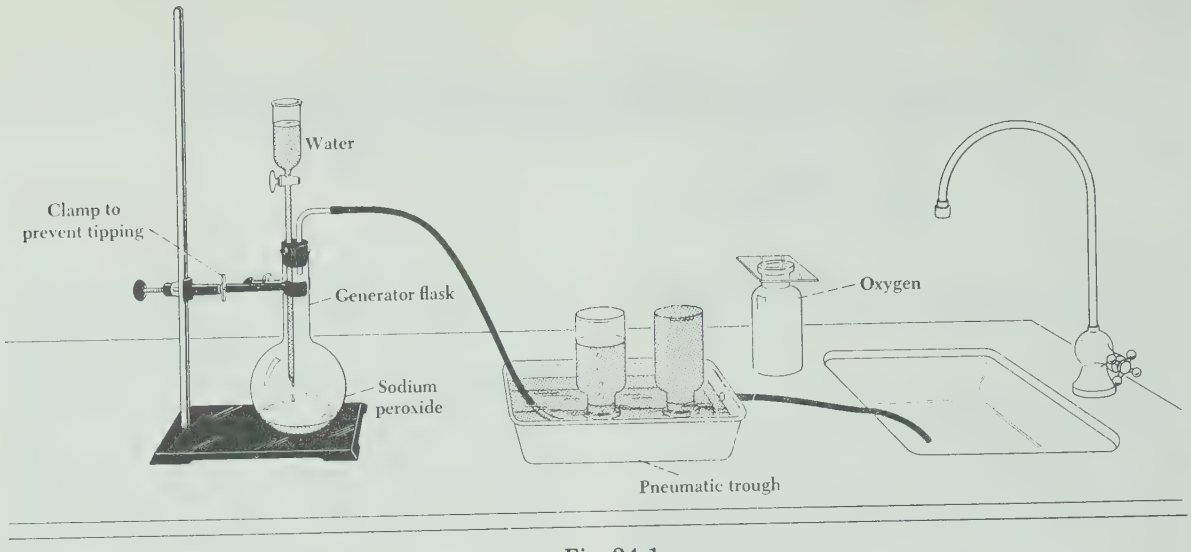


Fig. 24-1

tinue the addition of water to the generator until the reaction is completed.

Observations: Na_2O_2 is an oxidizing agent and could oxidize the paper. Wetting changes it into NaOH and O_2.

The first bottle of oxygen collected is impure, containing the displaced air in the generator.

The reaction in the generator is exothermic.

2. Clear away the oxygen-generating apparatus *after* the reaction has ceased. Select a 1-hole stopper to fit a hard glass test tube and insert the short leg of an 8 × 20 cm glass bend. Place a small coil of copper wire in the Pyrex test tubes Add 10 ml of distilled water to a second hand glass test tube. Cover the copper with 1 ml of water and add 4 ml of concentrated nitric acid. Immediately insert the stopper assembly and pass the evolved gas into the distilled water. The gaseous product is nitrogen dioxide, NO_2. **CAUTION:** *NO_2 is poisonous and must not be inhaled.* Describe it. What do you think is its source? After the evolution of the gas has subsided, heat the solution of NO_2, using a test tube clamp and a low flame. **CAUTION:** *Hold the test tube in a slanting position with the mouth of the tube turned away.* Move the tube over the flame to heat the solution uniformly and allow to boil quietly for about 1 minute. Set aside to cool for later use.

Observations: NO_2 is a reddish-brown gas. It is produced by the oxidation of Cu by HNO_3.

$Cu + 4HNO_3 \rightarrow Cu(NO_3)_2 + 2H_2O + 2NO_2(g)$.

3. Clean a deflagrating spoon, line it with asbestos paper, and heat it strongly for a moment in the burner flame. When cool, add a small lump of sulfur (no larger than a pea) and ignite over the burner. Quickly insert the burning sulfur well down into a bottle of oxygen. Keep the bottle covered as completely as possible with the glass plate. What is the product? When the combustion has ceased, remove the spoon (hot) and immediately add 10 ml of water to the bottle. Cover and shake vigorously; then set aside. Label to avoid confusion later.

Clean the deflagrating spoon as before; reline and preheat to burn away any combustible residue. Repeat the above procedure, using a small lump of red phosphorus (½ pea size) and another bottle of oxygen. Result? What is the combustion product? Does it appear to be soluble in water? Label and save. Repeat, using a small piece of charcoal and the remaining bottle of oxygen. What is this combustion product? Label and save.

Observations: The phosphorus burns very vigorously in the O_2 producing P_4O_{10} as a white cloud composed of solid small particles.

4. Place several test strips of red and blue litmus papers on a clean dry glass plate. Using a clean stirring rod, test each of the four solutions just prepared by dipping the stirring rod in the solution and then touching both red and blue test strips. *Rinse the rod in distilled water and wipe dry between tests.* Results? What action between water and nonmetallic oxides is indicated?

Divide each of the four solutions equally between 2 test tubes. Label all 8 test tubes to avoid confusion. To one test tube of each solution, add a drop of phenolphthalein indicator. To the remaining test tube of each solution, add 1 drop of methyl orange indicator. Shake each to mix uniformly. Observe the colors by looking lengthwise through the tubes against a white background. Results? Construct a suitable table and record your observations for Part 4. If the tests on the solutions containing CO_2 are indecisive, suggest a possible reason.

5. Place 4 test tubes in a beaker of cold water and add respectively 5 ml of dilute (approximately 3 *N*) H_2SO_4, H_3PO_4, HCl, and $HC_2H_3O_2$. Brighten 1-cm^2 pieces of sheet zinc and drop 1 square in each test tube. List the acids in the order of the activity observed.

Repeat this procedure with the 4 test tubes placed in a beaker of hot water. How does the action compare? Explain the difference observed.

Observations: HCl & H_2SO_4 react at about the same rate; H_3PO_4 less vigorously and $HC_2H_3O_2$ is the least reactive. The heated acids react more vigorously due to the increased rate of reaction at the higher temperatures.

QUESTIONS: 1. How can you define an acid anhydride? An acid anhydride is an oxide that reacts with water to form an acidic solution.

2. What general class of elements reacts with oxygen to form acid anhydrides? The oxides of nonmetals form acid anhydrides.

3. What is the effect of increased temperature on the chemical activity of acids? The acids are more active at higher temperatures.

FURTHER EXPERIMENTATION: Use a conductivity apparatus setup like the one shown on page 246 of MODERN CHEMISTRY, 1974 to test conductivity of glacial acetic acid. (Test with litmus paper and Hydrion paper.) To one sample of glacial acetic acid add an equal volume of benzene and stir the mixture. Test for conductivity. Repeat tests with litmus and Hydrion paper. Repeat with another sample of glacial acetic diluted with an equal volume of water and again test with litmus and Hydrion. Test a benzene solution of hydrogen chloride with litmus paper (dried in disiccator) and with Hydrion paper similarly dried. Test its conductivity. To this solution add an equal volume of water. Stopper and shake vigorously. Repeat the tests with litmus paper, Hydrion paper, and conductivity apparatus.

EQUATIONS

1. Reaction between sodium peroxide and water $2Na_2O_2 + 2H_2O \rightarrow 4NaOH + O_2(g)$
2. Reaction between sulfur dioxide and water $SO_2 + H_2O \rightarrow H_2SO_3$
3. Reaction between diphosphorus pentoxide and water $P_4O_{10} + 6H_2O \rightarrow 4H_3PO_4$
4. Reaction between carbon dioxide and water $CO_2 + H_2O \rightarrow H_2CO_3$
5. Reaction between nitrogen dioxide and water (nitrogen monoxide is one of the products). $3NO_2 + H_2O \rightarrow 2HNO_3 + NO(g)$
6. Reaction between zinc and sulfuric acid $Zn + H_2SO_4 \rightarrow ZnSO_4 + H_2(g)$
7. Ionization of acetic acid in water $HC_2H_3O_2 + H_2O \rightleftarrows H_3O^+ + C_2H_3O_2^-$
8. Ionization of hydrogen chloride in water $HCl + H_2O \rightarrow H_3O^+ + Cl^-$

NAME . . . . . PERIOD . . . . . DATE . . . . . .

INSTRUCTOR'S COMMENTS . . . . . .

Experiment 24 (b) Preparation and Properties of Hydroxides

PURPOSE: To prepare some hydroxides and to study the properties of the metallic hydroxides.

APPARATUS: Beaker, 250 ml; burner and tubing; clamp, buret; dish, evaporating; forceps; funnel; glass plates; graduated cylinder; ring, iron; ring stand; test tubes; watch glass; wire gauze.

MATERIAL: Barium hydroxide; calcium; calcium oxide; sodium; Hydrion paper D (pH:11-13); litmus papers, red and blue; filter paper; ammonia-water solution, concentrated; ammonia-water solution, dilute (1:4); hydrochloric acid, dilute (1:4); solutions of aluminum sulfate (0.03 *M*), iron(III) chloride (0.07 *M*); magnesium hydroxide (suspension); methyl orange; phenolphthalein; sodium hydroxide (2 *M*).

INTRODUCTION: A solution of a metallic hydroxide has basic properties due to the presence of hydroxide, OH^-, ions. Some very active metals, such as sodium and potassium, replace part of the hydrogen from water at ordinary temperatures to form solutions containing hydroxide ions. Such metallic oxides as calcium oxide, CaO, react directly with water to form basic solutions. Some insoluble hydroxides are formed by ionic reactions.

In the modern sense, a base is a proton acceptor. Thus many particles are basic. In general chemistry the most common base is the hydroxide, OH^-, ion. Soluble metallic hydroxides form our most common basic solutions.

CAUTION: *Do not handle sodium with the fingers.* Large pieces of sodium produce *dangerous explosions when placed on water.* Secure from your instructor a small piece of metallic sodium at the time you are ready to use it. You may use your forceps, or have the sodium placed on a piece of filter paper. Step back a few feet after you have added the sodium to water, since a slight explosion could occur near the end of the reaction.

PROCEDURE: 1. Read the *caution* before you begin. Drop a small piece of sodium, not larger than one quarter of a *small pea*, into an evaporating dish half-filled with water. Cover immediately with a glass plate. When the action is complete, test the solution with red and blue litmus papers. To make this test, place the test strips on a clean, dry glass plate. Then dip a clean stirring rod into the solution and touch the test strips. Result? Test the solution with Hydrion paper in the same manner as that used with the litmus papers and record the pH.

Transfer 2 ml of the solution to a test tube and add to it a drop of phenolphthalein solution. Result? Repeat with a second 2-ml portion and a drop of methyl orange solution. Result? To what particles in the solution do you attribute these color changes? Evaporate 3 ml of the solution to dryness on a watch glass over boiling water. Name the solid residue. *It is caustic and burns the flesh.*

Drop a small calcium turning into a test tube half-filled with water. After the reaction is complete, test the contents with red and blue litmus paper by dipping a stirring rod into the solution and applying to the litmus papers. Note how the test tube contents differ in appearance from the results obtained from the racetion of sodium with water. Test the solution with Hydrion paper, using the same technique as that used with litmus paper. Record the pH of the solution.

Observations: The red litmus turns blue. The blue litmus stays blue. Hydrion paper indicates a pH of 13. Phenolphthalein turns red and methyl orange turns yellow. The color changes are due to the presence of the OH^- ion. The solution feels slippery.

2. Test dry calcium oxide with litmus. Result? To one gram of *fresh* calcium oxide in a test tube, add 10 ml of water. Describe the action. What is this process called? Shake the tube for 2 or 3 minutes, then filter. Test the filtrate with litmus papers and Hydrion paper in the manner described in Part 1. Record the pH of the solution. Add a drop of phenolphthalein to one portion of the filtrate. Explain the result. Add a drop of methyl orange to another portion. Result? Name the responsible particles in

this solution. How would you describe the solubility of the product?

Observations: Dry CaO does not affect litmus paper. CaO reacts with water exothermically, swelling to a white powdery mass with the formation of $Ca(OH)_2$. This reaction is called slaking. Phenolphthalein turns red, litmus turns blue. The ion in solution is the OH^- ion. $Ca(OH)_2$ is only slightly soluble. The pH is approximately 12.

3. Test dry barium hydroxide with litmus. Result? To 0.5 g of barium hydroxide in a test tube, add 10 ml of water. Shake for 2 or 3 minutes, filter, and then test the filtrate with litmus papers, Hydrion paper, phenolphthalein, and with methyl orange as before. Record the pH. How do you interpret the results of these tests?

CAUTION: *Barium hydroxide is poisonous.*

Observations: Dry $Ba(OH)_2$ gives no reaction with litmus. Its filtered solution turns litmus blue, phenolphthalein red and has a pH of approximately 13. The OH^- ion is responsible for the indicator changes.

4. To 5 ml of magnesium hydroxide (suspension) in a test tube, add 10 ml of water. Shake for 2 or 3 minutes, filter, and then test the filtrate with litmus papers. Hydrion paper, phenolphthalein, and with methyl orange as before. Record the pH. How do you interpret the results of these tests?

Observations: The $Mg(OH)_2$ filtrate turns litmus blue, phenolphthalein pink-red, and the pH is approximately 11.

5. Add a few drops of concentrated ammonia-water solution to 10 ml of water in a test tube. Test this solution with litmus papers, Hydrion paper, phenolphthalein, and methyl orange. Result? Explain. Clamp the test tube in an inclined position and heat about 5 ml of the solution to boiling. Hold a piece of moist red litmus paper in the vapor escaping from the mouth of the tube, taking care that it does not touch the sides of the tube. Result? After the liquid has been boiled for a few minutes, test it again with red litmus (stirring rod technique). Result? Explain.

Observations:

Litmus turns blue; phenolphthalein pink-red; methyl orange to yellow, Hydrion paper pH approximately 11. These test results indicate presence of OH^- ion. During the heating NH_3 escapes from solution and causes the moist red litmus paper to turn blue. The boiled solution should not turn litmus blue, but some students may find that some NH_3 still remains in solution

6. Fill a test tube one-fourth full of a solution of iron(III) chloride, and add to it 2 or 3 ml of dilute ammonia-water solution. Shake the test tube to mix its contents. Name and describe the precipitate.

Observations:

Red-brown precipitate of iron(III) hydroxide forms.

7. Fill a test tube one-fourth full of a solution of aluminum sulfate, and add to it 2 or 3 ml of dilute ammonia-water solution. Shake. Name and describe the gelatinous precipitate formed. This precipitate does not settle readily and may be regarded as a suspension. Transfer one-half of the suspension to another test tube and add dilute hydrochloric acid with mixing until an excess is present. (How can you tell when this condition exists?) Result? Write the formula for the gelatinous precipitate (suspension) which is suitable to this behavior. To the portion of the suspension still in the original test tube, add a solution of sodium hydroxide, a few milliliters at a time, until an excess is present. Mixing may be necessary. Result? Explain. Can you write the formula of the original gelatinous precipitate in a form more suitable to show its behavior in the presence of a strong hydroxide solution? What name is used to describe this property in such substances?

Observations:

The gelatinous white precipitate is aluminum hydroxide. Excess HCl is determined by the use of litmus paper and the solution of the $Al(OH)_3$.

Mixing the remainnig half of the $Al(OH)_3$ precipitate with excess NaOH results in its solution since $Al(OH)_3$ is amphiprotic. It may be written as H_3AlO_3.

QUESTIONS: 1. What are two general methods of preparing soluble hydroxides?

(a) Active metal + water → soluble hydroxide (and hydrogen)
(b) Metallic oxide + water → soluble hydroxide.

2. Given an unknown solution, how would you test to determine whether it is acidic or basic? Test the solution with litmus paper. If it turns the test strip blue, it is basic; if it turns the test strip red, it is acidic.

3. What particle do the solutions of the soluble metallic hydroxides have in common?

Soluble metal hydroxides produce solutions which have basic properties due to the presence of the hydroxide ion.

4. Based on the results obtained by use of the Hydrion paper and the color code for the corresponding pH, list the four soluble hydroxides in order of *decreasing* hydroxide

ion concentration. NaOH, $Ba(OH)_2$, $Ca(OH)_2$, $Mg(OH)_2$.

5. How does the strength of the hydroxide of Na (Group I) compare with that of the hydroxides of Ba, Ca, Mg (Group II)?

The hydroxides of Group I are strong hydroxides, while those of Group II are, with the exception of $Ba(OH)_2$, weakly basic due to their slight solubility.

6. Based on your pH observations how does the strength of the hydroxides of Group II metals vary with the ionic size?

The strength of the hydroxides of Group II metals increases with increasing ionic size.

7. Based on your pH observations, which metallic hydroxide is approximately as basic as the diluted ammonia-water?

$Mg(OH)_2$.

FURTHER EXPERIMENTATION: Test the solutions of NaOH, $Ba(OH)_2$, $Ca(OH)_2$, and $Mg(OH)_2$ with the conductivity apparatus, using a 60-watt bulb. For the one(s) which does (do) not light this bulb, try a 25-watt bulb.

Test the solution of NH_3 in benzene with litmus papers and Hydrion paper indicators. Also test conductivity again. Repeat after shaking the benzene solution with an equal volume of distilled water.

EQUATIONS: Write in ionic form.

1. Reaction between sodium and water $2Na + 2H_2O \rightarrow 2NaOH + H_2(g)$
2. Reaction between calcium and water $Ca + 2H_2O \rightarrow Ca(OH)_2 + H_2(g)$
3. Reaction between calcium oxide and water $CaO + H_2O \rightarrow Ca(OH)_2$
4. Reaction between ammonia and water $NH_3 + H_2O \rightleftarrows NH_4^+ + OH^-$
5. Reaction between iron(III) chloride solution and ammonia-water solution $FeCl_3 + 3NH_3 + 3H_2O \rightarrow Fe(OH)_3(s) + 3NH_4Cl$
6. Reaction between aluminum sulfate solution and ammonia-water solution. $Al_2(SO_4)_3 + 6NH_3 + 6H_2O \rightarrow 2Al(OH)_3(s) + 3(NH_4)_2SO_4$
7. Reaction between aluminum hydroxide and hydrochloric acid solution $Al(OH)_3(s) + 3H_3O^+ \rightarrow Al(H_2O)_6^{+++}$ or $Al(OH)_3 + 3HCl \rightarrow AlCl_3 + 3H_2O$
8. Reaction between aluminum hydroxide and sodium hydroxide solution $Al(OH)_3(s) + OH^- \rightarrow AlO_2^- + 2H_2O$ or $Al(OH)_3 + NaOH \rightarrow NaAlO_2 + H_2O$

Experiment 25

NAME . . . . . PERIOD . . . . . DATE . . . . . .

INSTRUCTOR'S COMMENTS . . . . . .

Hydronium Ion Concentration, pH

PURPOSE: To determine the pH of various solutions of comparable concentration, and to study the effect of dilution on the pH of acids and a metallic hydroxide.

APPARATUS: Beaker, 250 ml; graduated cylinders, 10 ml, 50 ml; test tubes.

MATERIAL: pH papers, wide range, i.e. A & B, narrow range, i.e. D, Hydrion papers or equivalent pH papers; solutions of acetic acid (0.10*M*); ammonia water (0.10*M*); ammonium acetate (0.10*M*); hydrochloric acid (0.10*M*); phosphoric acid (0.033 *M*); sodium carbonate (0.050 *M*); sodium chloride (0.10 *M*); sodium hydrogen carbonate (0.10 *M*); sodium hydroxide (0.10 *M*).

INTRODUCTION: The concentration of hydronium ion is usually represented by the expression $[H_3O^+]$ which means the hydronium ion concentration in moles (gram-ions) per liter of solution. A one molar (1.0-*M*) solution of hydronium ions contains 1 mole, 19 g, of hydronium ion per liter of solution.

Pure water is slightly ionized and contains 0.0000001 mole of hydronium ion per liter. Its hydronium ion concentration, $[H_3O^+]$ is therefore represented as 1.0×10^{-7} *M* or 10^{-7} *M*. Since pure water is neutral, its hydroxide ion concentration is also 1.0×10^{-7} *M* or 10^{-7} *M*. In any water solution, the product of the hydronium and hydroxide ion concentrations is known as the ion-product of water and is equal to 1.0×10^{-14}. This equilibrium is represented by the equation:

$$H_2O + H_2O \leftrightharpoons H_3O^+ + OH^-$$

For neutral water, (25°C)

$$K_w = 10^{-14} = [H_3O^+] \times 10^{-6}$$
$$= (1 \times 10^{-7})(1 \times 10^{-7}) = 1 \times 10^{-14}$$

The pH of a solution is defined as the common logarithm of the reciprocal of the hydronium ion concentration. For neutral water,

$$pH = \log \frac{1}{0.0000001} = \log \frac{1}{10^{-7}}$$

Therefore: pH = log 10^7, and pH = 7.

If the hydronium ion concentration is greater than that of pure water, the pH of the solution is numerically less than 7. Thus, for a solution which is acid such as 0.000001-*M* (1×10^{-6}-*M*) HCl (completely ionized), the pH is 6. For acidic solutions, therefore, the pH is *smaller* than 7. The smaller the pH the larger the hydronium ion concentration. If the solution is basic,

DATA TABLE 1

0.1-*N* Solutions	Indicator color	Numerical pH	Strength as acid or base
HCl	Student Answers	1	strong acid
$HC_2H_3O_2$		3	weak acid
H_3PO_4	↓	1.5	weak acid, stronger than acetic
NaOH		13	strong base
NH_3		11	weak base
NaCl	↓	7	neutral
Na_2CO_3		11+	weak base, stronger than NH_3
$NaHCO_3$		8+	weak base, weaker than Na_2CO_3
$NH_4C_2H_3O_2$	↓	7	neutral

its hydronium ion concentration is less than 10^{-7} *M* and its pH is *larger* than 7. For a 1×10^{-6}-*M* solution of NaOH (completely dissociated), the hydroxide ion concentration is 1×10^{-6}. Its hydronium ion concentration therefore is 1×10^{-8}, and its pH is 8.

$K_w = 10^{-14} = [H_3O^+] \times 10^{-6}$
$[H_3O^+] = 10^{-8}$; the pH being 8

SUGGESTIONS: The teacher may wish to demonstrate the use of a pH meter to complement the use of the indicator and to obtain more exact pH readings.

PROCEDURE: 1. Test for the pH of each of the solutions by dipping a stirring rod into each of the solutions and applying first to the wide range pH papers (A and B) and if necessary, to the narrow (short) range paper (D). Record your observations and complete Data Table 1.

2. *Effect of dilution on pH of acids and hydroxides.* Start with 5.0 ml of stock (0.1-*M*) solutions and dilute to 50 ml. Save 5 ml of the diluted solution in a properly labeled test tube and use another 5.0 ml for the next dilution to 50 ml. Repeat the dilution process 4 times for each stock 0.1-*M* solution, in each case saving the 5.0 ml sample for testing with the pH papers as in Part 1. Record your results in Data Table 2.

QUESTIONS: 1. Which of the substances among those having a definite acidic reaction is (a) the strongest acid, (b) the weakest acid? (a) HCl

(b) $H_2C_2H_3O_2$

2. Why is a 0.1-*M* solution of NaOH a much stronger base than a 0.1-*M* solution of NH_3-aq?

NaOH is completely dissociated. The NH_3(aq) is only slightly ionized.

3. What effect does dilution have on the pH of (a) an acid, (b) a base?

(a) The pH of an acid is increased. (b) The pH of a base is decreased.

DATA TABLE 2

HCl	Calculated pH	Indicator color	Observed pH
1. 0.1 *M*	1	Student Answers	Student Answers
2. 0.01 *M*	2	↓	↓
3. 0.001 *M*	3		
4. 0.0001 *M*	4		
5. 0.00001 *M*	5		

NaOH	Calculated pH	Indicator color	Observed pH
1. 0.1 *M*	13	Student Answers	Student Answers
2. 0.01 *M*	12	↓	↓
3. 0.001 *M*	11		
4. 0.0001 *M*	10		
5. 0.00001 *M*	9		

$HC_2H_3O_2$	Indicator color	Observed pH
1. 0.1 *M*	Student Answers	Student Answers
2. 0.01 *M*	↓	↓
3. 0.001 *M*		
4. 0.0001 *M*		
5. 0.00001 *M*		

Experiment 26

NAME. . . . . PERIOD . . . . . DATE. . . . . .

INSTRUCTOR'S COMMENTS. . . . . .

Brønsted Acids and Bases, Indicator

PURPOSE: To show the existence of conjugate acid-base pairs in protolytic reactions involving a weak acid or a weak base, and to show how the relative strength of an acid or base causes indicator reactions.

APPARATUS: Burner and tubing; clamp, test tube; graduated cylinder; test tubes.

MATERIAL: Acetic acid, glacial; ammonium chloride, crystals; sodium acetate, pellets; litmus papers, red and blue; acetic acid (1.0 *M*); ammonia-water solutions (conc., 1.0 *M*); hydrochloric acid (1.0 *M*); sulfuric acid (1.5 *M*); solutions of ammonium chloride (1.0 *M*); sodium acetate (1.0 *M*); sodium carbonate (0.05 *M*); sodium chloride (1.0 *M*); sodium hydroxide (6 *M*, 0.1 *M*); ammonia, household, bromthymol blue; lemon juice; vinegar.

INTRODUCTION: According to the Brønsted concepts, an acid is a proton donor and a base is a proton acceptor. When an acid gives up a proton, the remainder of the acid particle becomes itself capable of accepting a proton. This remainder is called the conjugate base of the acid. Together they constitute a conjugate acid-base pair. This pair consists of two particles differing from each other by a proton. If HB represents a Brønsted acid, the following shows the relationships of the conjugate acid-base pair: A_1 and B_1

$$\underset{}{HB} + H_2O \rightleftarrows \underset{A_1}{H_3O^+} + \underset{B_1}{B^-}$$

The protolytic reaction contains a second conjugate-acid base pair: A_2 and B_2

$$\underset{}{HB} + \underset{B_2}{H_2O} \rightleftarrows \underset{A_2}{H_3O^+} + B^-$$

For a weak acid, this reaction proceeds to the right to a limited extent only. An indicator is a colored substance, generally a dye, which changes color in the presence of an acid or a base. It may be regarded as a weak acid, represented by the formula HIn. In a water solution of the indicator at equilibrium, the situation may be represented by the equation

$$HIn + H_2O \rightleftarrows H_3O^+ + In^-$$

A simpler version of this is

$$HIn \rightleftharpoons H^+ + In^-$$

The molecular species, HIn, has one color and the anion, In^-, has a different color. At equilibrium, the characteristic color is that resulting from the proportions of these two species, HIn and In^-, mixed in the solution. Since the indicator is a weak acid, its ionization is slight. The equilibrium color in neutral water depends on the relative concentrations of HIn and In^-. The ionization constant for the indicator, K_a like that of other weak acids (refer to Chapter 20, MODERN CHEMISTRY, 1974 for detailed discussion) is represented by the expression:

$$K_a = K_{HIn} = \frac{[H^+]\,[In^-]}{[HIn]}$$

Increasing the hydronium ion concentration, represented simply as $[H^+]$, by the addition of acid causes the formation of more of the molecular species, HIn, thereby decreasing the concentration of the In^- ion. Decreasing the hydronium ion concentration, for example by the addition of OH^- ion, results in an increase in the concentration of the In^- ion and consequent decrease in the concentration of HIn. Thus the ratio $[In^-]/[HIn]$ is inversely proportional to the hydronium ion concentration. The indicator end point is the hydronium ion concentration (pH) at which a perceptible change in indicator color occurs. The definite range of pH values, over which the color of the indicator changes, is called the transition interval. (Consult Table of Indicator Colors, Chapter 15, and the color plates, in MODERN CHEMISTRY, 1974.)

PROCEDURES: 1. Remove the stopper from a bottle of glacial acetic acid and carefully test for the odor of acetic acid using the wafting technique, Fig. F-5. Place one drop of the glacial acetic acid on a piece of blue litmus paper. Result? Place this litmus paper into 2 ml of water in a test tube. Result? What ion is present in the solution? Complete the equation for this ionization in the Data Table. Add 6 pellets of sodium acetate to 2 ml of water in a test tube. Test for its odor. Sodium acetate is a salt. Show the ions present in its solution by complet-

DATA TABLE 1

	Acetic acid (glacial)	Ammonia-water solution
Odor	sour	penetrating
Litmus reaction	none	
a. H_2O added	blue litmus turns red	red litmus turns blue
b. Ion identified	H_3O^+	OH^-
c. Ionization equation	$HC_2H_3O_2 + H_2O \rightleftarrows H_3O^+ + C_2H_3O_2^-(aq)$	$NH_3 + H_2O \rightleftarrows NH_4^+(aq) + OH^-(aq)$
	Sodium acetate	**Ammonium chloride**
Odor	none	none
Dissociation equation	$NaC_2H_3O_2(s) \rightarrow Na^+(aq) + C_2H_3O_2^-(aq)$	$NH_4Cl(s) \rightarrow NH_4^+(aq) + OH^-(aq)$
and H_2SO_4		and NaOH
a. Odor	sour odor (acetic acid)	penetrating odor of NH_3
b. Net ionic equation	$C_2H_3O_2^-(aq) + H_3O^+(aq) \rightarrow HC_2H_3O_2 + H_2O$	$NH_4^+ + OH^- \rightarrow NH_3(g) + H_2O$

ing the equation in Data Table 1. To six pellets of solid sodium acetate add 2 ml of 1.5-*M* H_2SO_4. Heat gently. Identify the odor of one of the products. Complete the representative net ionic equation in the Data Table.

Remove the stopper from the bottle of concentrated ammonia-water solution. Carefully test for the odor of ammonia using the wafting technique, Fig. F-5. Place a drop of the solution on a piece of red litmus paper. Result? What ion is present in the solution? Complete the equation for this ionization in the Data Table. Dissolve 6 crystals of ammonium chloride in 2 ml of water and test for its odor. Ammonium chloride is a salt. Show the ions present in its solution by completing the representative equation in the Data Table. Add 2 ml of 6-*M* sodium hydroxide solution, gently heat the test tube, and test for odor using the wafting technique. Complete the representative net ionic equation for the reaction.

2. Add 5 drops of bromthymol blue to 2.0-ml samples of the solutions listed. Compare indicator colors by looking through the tube lengthwise against a white (paper) background. Report the indicator colors in Data Table 2. Save solutions *a* and *b*. Mix them and note the resultant color.

DATA TABLE 2

Solutions	Color of indicator
0.1-*M* HCl *(a)*	yellow
0.1-*M* NaOH *(b)*	blue
0.1-*M* NaCl	green
vinegar	yellow
lemon juice	yellow
ammonia (household)	blue
0.05-*M* Na_2CO_3	blue
(a) and *(b)* mixed	green

3. In this part of the experiment you will determine the comparative strengths of the weak acids: acetic acid, the molecular form of bromthymol blue represented by HBtB, and ammonium ion. The equations for the conjugate acid-base pairs in water solution are:

$$\begin{array}{cccccc} A_1 & B_2 & A_2 & B_1 \\ HC_2H_3O_2 & + H_2O & \leftrightarrows H_3O^+ & + C_2H_3O_2^- \\ H_2O & + NH_3 & \leftrightarrows NH_4^+ & + OH^- \\ HBtb & H_2O & \leftrightarrows H_3O^+ & + Btb^- \end{array}$$

(a) In a test tube mix 2 ml of 1.0-*M* acetic acid with 2 ml of 1.0-*M* sodium acetate. This solution has equal concentrations of acetic acid and its conjugate base, the acetate ion. What is the concentration of each of these in the mixture? Add 5 drops of bromthymol blue to the mixture. Result? In which of its colored forms is the indicator in this mixture? Complete the equation in Data Table 3 to show how this result took place. Which is the stronger acid, acetic acid or bromthymol blue indicator?

(b) Mix 2 ml of 1.0-*M* NH_3-aq with 2 ml of 1.0-*M* NH_4Cl. This solution contains equal concentrations of NH_3 and its conjugate acid, NH_4^+. What is the concentration of each of these in the mixture? Add 5 drops of bromthymol blue to the mixture. Result? In which of its forms is the indicator present? Complete the equation in Data Table 3 to show how this result took place. Complate Data Table 3 below.

QUESTIONS: 1. Of the three acid species being evaluated, which is (a) the strongest acid? $HC_2H_3O_2$ (b) the weakest acid? NH_4^+

2. Of the conjugate bases, which is (a) the strongest base? NH_3 (b) the weakest base? $C_2H_3O_2^-$

DATA TABLE 3

	$HC_2H_3O_2$	NH_3-aq
Concentrations of conjugate acid-base pair	0.05*M*	0.05*M*
Color after indicator addition	yellow	blue
Form of indicator present	HBtb	Btb^-
Equations	$HC_2H_3O_2$ + HBtb + $Btb^- \rightarrow$ $C_2H_3O_2^-$	NH_3 + HBtb $\rightarrow$ NH_4^+ + Btb^-

NAME . . . . . PERIOD . . . . . DATE . . . . . .
INSTRUCTOR'S COMMENTS . . . . . .

Experiment 27 Relative Strengths of Acids and Bases

PURPOSE: To develop a table of the comparative strengths of a series of weak acids and, conversely, a table of the strengths of their conjugate bases.

APPARATUS: Graduated cylinder; test tubes.

MATERIAL: Acetic acid (0.1 *M*); boric acid (0.1 *M*); carbonic acid (sat. CO_2 sol'n); hydrochloric acid (0.1 *M*); hydrosulfuric acid (sat. sol'n of H_2S); oxalic acid (0.1*M*); solutions of aluminum chloride (0.1 *M*); ammonium chloride (0.1 *M*); calcium nitrate (0.05 *M*); sodium hydrogen carbonate (0.1 *M*); sodium hydrogen sulfate (0.1 *M*); sodium oxalate (0.05 *M*); indicators: bromthymol blue; litmus; methyl orange; phenolphthalein.

INTRODUCTION: All strong acids have approximately the same acid strength in water solution since they are highly ionized. Their protolytic reaction with water is practically complete even in concentrated solutions. Hydrochloric acid is used as a representative strong acid. In aqueous solutions of weak acids the concentration of the hydronium ion is quite low even in dilute solutions. The weak acid does not compete very successfully with hydronium ions to donate protons to a base. Conversely, the water molecules do not compete very successfully with the conjugate base of the weak acid to accept protons. Thus, in the reversible situation, the reaction toward the weaker acid and base predominates. The situation is represented by the generalized equation:

weaker	weaker		stronger	stronger
HB	H_2O	$\rightleftarrows$	H_3O^+	B^-
acid	base		acid	base

PROCEDURE: 1. Add one drop of each indicator to a separate 2-ml sample of each of the solutions listed in the Data Table. Estimate each color by looking through the vertical test tube against a white back-

TABLE OF INDICATOR COLORS

Indicator	Notation	Color			Transition interval (pH)
		Acid	Transition	Alkaline	
Methyl orange	HM	red	orange	yellow	3.2-4.4
Bromthymol blue	HBtb	yellow	green	blue	6.0-7.6
Litmus	HL	red	pink	blue	5.5-8.0
Phenolphthalein	HP	colorless	pink	red	8.2-10.0

DATA TABLE

0.1-*M* solutions	Methyl orange	Bromthymol blue	Litmus	Phenolphthalein	Estimated pH
HCL	red	yellow	red	colorless	<3.2
HSO_4-($NaHSO_4$)	red	yellow	red	colorless	<3.2
H_2CO_3 (sat. soln.)	red	yellow	red	colorless	<3.2
$H_2C_2O_4$	red	yellow	red	colorless	<3.2
H_2S (sat. soln.)	red	yellow	red	colorless	<3.2
$Al(H_2O)_6^{+3}$ ($AlCl_3$)	red	yellow	red	colorless	<3.2
$HC_2H_3O_2$	red	yellow	red	colorless	<3.2
HCO_3-($NaHCO_3$)	yellow	blue	blue	pink-red	>8.4
NH_4^+(NH_4Cl)	red	yellow (green)	pink	colorless	<3.2

ground. Record each color and the estimated pH of the solution in the Data Table. Save the first set with HCl for color comparison purposes. Wash out the test tubes thoroughly with distilled water before proceeding to the next set.

Arrange the solutions in order of *decreasing* acid strength. Next to each of the acids, write the formula for its conjugate base. Treat each polyprotic species as if it relinquishes one proton only.

Repeat the indicator tests with 2-ml samples of 0.1-*M* boric acid (H_3BO_3). Results? What is the approximate pH of its solution? Where does boric acid fit into your table? For which of the indicators is its acid (molecular) form a stronger acid than boric acid?

Observations: Methyl orange: yellow; Bromthymol blue; yellow; Litmus: pink-red; Phenolphthalein: colorless. Approximate pH is between 4.4 and 6.0. Between NH_4^+ and HCO_3^-. Methyl orange is stronger than boric acid.

2. To 5 ml of 0.05-*M* calcium nitrate solution add 5 ml of 0.05-*M* sodium oxalate solution. Result? Complete the representative formula equation below. Divide the test tube contents into two equal portions. To one portion add 5 ml of 0.1-*M* HCl. Result? To the second portion add 5 ml of 0.1-*M* $HC_2H_3O_2$. Result? What do these results show about the relative strength of the three acids: hydrochloric, acetic, and oxalic?

$$Ca(NO_3) + Na_2C_2O_4 \rightarrow \ldots\ldots$$

$2\,NaNO_3 + CaC_2O_4(s)$

Observations: The precipitate dissolves in HCl but does not dissolve in $HC_2H_3O_2$. The relative strengths are $HCl > H_2C_2O_4 > HC_2H_3O_2$.

RELATIVE STRENGTHS OF ACIDS AND BASES

Acid formula	Conjugate base formula
HCl	Cl^-
$H_2C_2O_4$	$HC_2O_4^-$
HSO_4^-	SO_4^{--}
$HC_2H_3O_2$	$C_2H_3O_2^-$
$Al(H_2O)_6^{+3}$	$Al(H_2O)_5(OH)^{+2}$
H_2CO_3	HCO_3^-
H_2S	HS^-
NH_4^+	NH_3
HCO_3^-	CO_3^{--}

Decreasing Acid Strength ⟵ ⟶ Decreasing Base Strength

Note: Students will probably have a variety of answers. It is suggested that the instructor demonstrate this order by use of pH meter.

Experiment 28

NAME . . . . . PERIOD . . . . . DATE . . . . . .

INSTRUCTOR'S COMMENTS . . . . . .

Titration of an Acid and a Hydroxide, Heat of Neutralization

PURPOSE: To determine the concentration of a basic solution expressed in normality, and the heat of neutralization.

APPARATUS: Two beakers, 150 ml; 2 burets; calorimeter, plastic cups; clamp, double buret; flask, Erlenmeyer; graduated cylinder, 50 ml; ringstand; thermometer; wash bottle.

MATERIAL: Solutions of hydrochloric acid (3.0*M*, standard solution about 0.1 *N*); solutions of sodium hydroxide (3.0 *M*, ? *N*); phenolphthalein.

INTRODUCTION: Titration is a process by which the concentration of a solution is determined by its reaction with a standard solution, the concentration of which is precisely known. The process consists of the gradual addition of the standard solution to a measured quantity of the solution of unknown concentration until the same number of gram-equivalent weights (equivalents) of each solute has been used. The point in the titration at which equal equivalents are present is known as the end point. An indicator is used to detect the end point in the process.

An indicator which undergoes a color change in the pH region of the end point of the reactants should be selected. In this experiment, the end point will be reached at a pH about 7. Litmus is a suitable indicator. Where the precision is not too exacting, little difference will be noticed if phenolphthalein is chosen instead of litmus. Phenolphthalein is a somewhat more suitable indicator for beginning students.

The concentration of solutions in titration is commonly expressed in *normality*. The advantage of normality over molarity (formality) or molarity is that equal volumes of solutions of the same normality are always chemically equivalent. If solution No. 1 has a higher normality than solution No. 2, a smaller volume of No. 1 will be required to reach an end point with a given volume of No. 2. *That is, the relative volumes are inversely proportional to the normalities of the solutions when equal equivalents of both have reacted:*

$$\frac{N_1}{N_2} = \frac{V_2}{V_1}$$

The normality of the standard solution and the volumes of both solutions are known when the end point of the titration is reached. Therefore, the normality of the solution of unknown normality can be found:

$$N_2 = \frac{N_1 V_1}{V_2}$$

Neutralization is the reaction between hydronium ions and hydroxide ions to form water. In a broader sense, acid-base neutralization is the reaction that occurs when equivalent quantities of an acid and a base are mixed. The meat of neutralization in kilocalories per mole (kcal/mole) is the heat liberated when one mole of water is formed by the reaction between one mole of hydronium ions and one mole of hydroxide ions. This may be expressed by a thermochemical equation (See Chap. 20, Modern Chemistry, 1974): H_3O^+ (aq) + OH^-, (aq) → 2 H_2O(l) + heat of neutralization, or ,more simply as H^+(aq) → OH^-(aq) → H_2O(l) + heat of neutralization.

SUGGESTIONS: Reagent grade chemicals should be used in preparing the solutions. A fresh bottle of hydrochloric acid should be used and preferably one which shows the actual assay of HCl rather than the average assay. If one laboratory session is to be allowed for this experiment, the standard acid solution and the basic solution of unknown normality should be prepared in advance by the instructor. If two sessions are available, the first could be used profitably by allowing the students to prepare their own standard solutions. The assay (in % HCl) and the specific gravity of the acid should be given to the students. Small volumetric flasks should be issued for this work.

The concentration of the sodium hydroxide solution should be roughly the same as that of the standard acid and should be precisely known by the instructor as a check against the preparation of the standard solutions by the students.

PROCEDURE: 1. Set up apparatus as shown in Fig. 28-1. Take approximately 100 ml each of hydrochloric acid and sodium hydroxide separately in clean, dry, labeled 150-ml beakers. Pour directly from the beaker into one buret a 10-ml portion of sodium hydroxide solution. Rinse the walls of the buret thoroughly with this solution, allow it to drain through the stopcock, and discard it. Rinse the buret two more times in similar fashion using a new 10-ml portion of sodium hydroxide each time. Discard all rinsings. Then fill the buret with sodium hydroxide solution above the zero mark. Withdraw enough solution to remove the air from the jet tip and bring the liquid level into the graduated region of the buret. In similar fashion rinse and fill the acid buret.

2. Record the initial reading of each buret, estimating to the nearest 0.01 ml, in the Data Table 1. Then draw off about 15 ml of the hydroxide into an Erlenmeyer flask and add approximately 25 ml of distilled water to give volume to the solution. Add one or two drops of phenolphthalein solution as an indicator. Now run in the acid solution *slowly*, stopping occasionally to mix, using a swirling motion. See Fig. F-13. Wash down the inside surface of the flask frequently with a little distilled water, using your wash bottle. Why may distilled water be added without disturbing the titration procedure? When the pink color of the solution begins to disappear at the point of contact with the acid, add the acid *drop by drop*, swirling the flask gently after each addition, until the last drop added causes the color to disappear. A sheet of white paper under the flask makes it easier to detect the color change. Now add the basic solution dropwise to just return the color. Go back and forth over the end point several times until one drop of the basic solution just brings out a faint pink color. Wash down the inside surface of the flask and make dropwise additions if necessary to re-establish the faint pink end-point color. Read the burets, estimating to the nearest 0.01 ml, and record in Data Table 1.

Repeat the titration for a second trial, and if time permits, a third trial.

3. Measure out 50 ml of 3.0-*M* HCl and pour into the calorimeter. Take the temperature of this solution. Measure out 50 ml of 3.0-*M* NaOH and pour into a beaker. Clean and dry the thermometer and take the temperature of the sodium hydroxide solution. Ideally, these solutions should have the same temperature. If they do not, permit them to stand until they do reach the same temperature, carefully cleaning and drying the thermometer each time it is used. Record this temperature in Data Table 2.

Pour the sodium hydroxide solution into the hydrochloric acid solution in the calorimeter. Replace the calorimeter cover, insert the thermometer and use it to gently stir the contents of the calorimeter. Record the maximum temperature reached while the neutralization is taking place.

DATA TABLE 1 Students answers.

	First trial	Second trial	Third trial
Initial reading, acid buret	ml	ml	ml
Final reading, acid buret	ml	ml	ml
Volume of acid used	ml	ml	ml
Initial reading, hydroxide buret	ml	ml	ml
Final reading, hydroxide buret	ml	ml	ml
Volume of hydroxide used	ml	ml	ml
Normality of standard acid	*N*	*N*	*N*
Normality of base (computed)	*N*	*N*	*N*
	Average value		*N*
Equivalents of NaOH in 100 ml of basic solution			/100 ml
Mass of NaOH in 100 ml of basic solution			g/100 ml

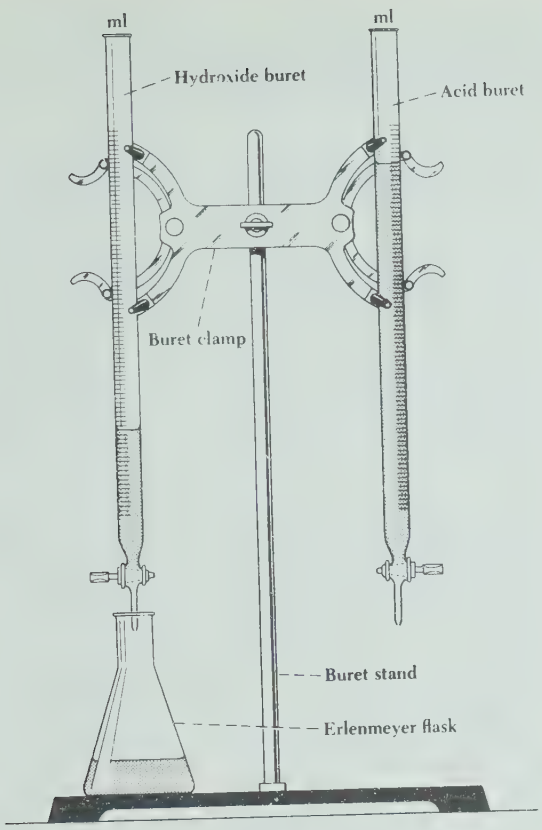


Fig. 28-1

CALCULATIONS: Titration **1.** The volumes of acid and hydroxide solutions used in each trial and the normality of the standard acid solution are known. Solve the inverse proportion as given in the Introduction to find the normality of the basic solution for each trial. Determine the *average value* of the normalities from the different trials and record each result in Data Table 1.

2. By definition:

$$\text{normality} = \frac{\text{no. equivalents solute}}{\text{no. liters solution}}$$

and

no. equivs. solute =
normality × no. liters sol'n

Knowing the normality (average value) of the sodium hydroxide solution, determine the number of equivalents of NaOH in 10$\bar{0}$ ml of solution.

3. Express the no. equivs/10$\bar{0}$ ml (as found above) in no. g NaOH/100-ml of solution, using the following relationship:

$$\frac{\text{no. equivs.}}{10\bar{0}\text{ ml}} \times \frac{\text{g NaOH}}{\text{equiv}} =$$

no. g NaOH/10$\bar{0}$ ml of solution

CALCULATIONS: Heat of Neutralization.

1. Upon ionization one mole of hydrochloric acid yields one mole of hydronium ions. Upon dissociation, one mole of sodium hydroxide yields one mole of hydroxide ions. Therefore *one* mole of each of these substances yields *one* gram-equivalent of their respective ions.

In this experiment, you used an identical number of moles of hydronium and hydroxide ions. Neutralization therefore produced this number of moles of water. By definition:

No. moles of ion(s) =
molarity × no. of liters sol'n.

Calculate the number of moles of each ion and record in Data Table 2.

Also record the number of moles of water produced.

2. The total volume of solution heated by the heat liberated during neutralization is equal to the *total* volume of the reactants. We may assume that its mass in grams is equal to its volume. Why? Enter this quantity in Data Table 2.

Multiply this mass by the temperature change and then by 1.0 cal/gC° to obtain the number of calories of heat liberated.

3. Calculate the heat of neutralization in kilocalories per mole (kcal/mole) using the relationship:

$$\text{Heat of neutralization (kcal/mole)} = \frac{\text{kilocalories liberated}}{\text{no. moles of water formed}}$$

Record the calculated heat of neutralization in Data Table 2.

DATA TABLE 2

Volume (3.0-*M* HCl)	ml
Volume (3.0-*M* NaOH)	ml
Temperature (3.0-*M* HCl)	°C
Temperature (3.0-*M* NaOH)	°C
Maximum temperature (during neutralization)	°C
Change in temperature	C°
Mass of solution heated	g
Moles H_3O^+	mole
Moles OH^-	mole
Moles H_2O formed	mole
Heat liberated	cal
Heat liberated	kcal
Heat of neutralization	kcal/mole

Experiment 29

NAME . . . . . PERIOD . . . . . DATE . . . . . .

INSTRUCTOR'S COMMENTS . . . . . .

Percentage of Acetic Acid in Vinegar

PURPOSE: To determine the acidity of vinegar, using the titration technique.

APPARATUS: Two beakers, 150 ml; 2 burets; clamp, double buret; flask, Erlenmeyer; ring stand; wash bottle.

MATERIAL: Vinegar; standard solution of sodium hydroxide (approximately 0.5 *N*); solution of phenolphthalein indicator.

INTRODUCTION: The quantity of acid in a sample of vinegar may be found by titrating the sample against a standard basic solution. Most vinegars on sale have a mass percentage of between 4.0% and 5.5% acetic acid. By determining the volume of sodium hydroxide solution of known normality necessary to neutralize a measured quantity of the vinegar, the normality of the vinegar can be calculated. From the normality, the mass percentage of acid present can then be calculated. Interesting results are obtained if the students bring samples of vinegar from home.

SUGGESTION: Review the discussion of titration technique and determination of the normality of the unknown solution presented in the Introduction of Experiment 28. Also review the computation methods carried out in the same experiment.

To remove color from a sample of cider vinegar, the instructor may filter it through boneblack before the class assemblies. This makes it easier to detect the end point of the titration.

As an alternate procedure the experiment may be performed by part or all of the glass using molarities instead of normalities. Change using molarities instead of normalities in the Data Table to molarities and the N(s) to M(s). Use Calculation 4 (optional) instead of Calculations 1-3.

PROCEDURE: 1. Transfer approximately 100 ml each of vinegar and sodium hydroxide solution to separate clean, dry, labeled 150-ml beakers. Pour directly from the beaker into one buret a 10-ml portion of the vinegar. Rinse the walls of the buret thoroughly with the vinegar, allow it to drain through the stopcock, and discard it. Rinse the buret two more times in similar fashion using a new 10-ml portion of vinegar each time. Discard all rinsings. Then fill the buret with vinegar above the zero mark. Withdraw enough solution to remove the air from the jet tip and bring the liquid level into the graduated region of the buret. In similar fashion rinse and fill the other buret with sodium hydroxide solution. Record the initial readings of both burets, estimating to the nearest 0,01 ml.

2. Withdraw about 15 ml of the vinegar into an Erlenmeyer flask. Add 50 ml of distilled water to the flask to add volume and to dilute any color present, thus making it easier to determine the color change when the end point is reached. Add one or two drops of phenolphthalein solution to serve as an indicator. Then titrate the vinegar with the standard solution of sodium hydroxide, swirling and washing down the flask frequently. Add the hydroxide solution drop by drop near the end of the operation, until the last drop (after swirling) just turns the solution a pink color. Now add successive quantities of both solutions dropwise, going back and forth over the end point, until the end point is clearly established. This is indicated by the slightest suggestion of pink coloration in the flask. The end point is most satisfactorily determined with the flask resting on a sheet of white paper and with a beaker of distilled water alongside for comparison purposes. Record the final buret readings of both solutions, estimating to the nearest 0.01 ml.

3. Discard the liquid in the flask, rinse thoroughly with distilled water, and run a second titration, proceeding as before. A third titration should also be made if time permits. Chemists like to have three consistent determinations before they feel confident that their titrations are reliable. Enter the buret readings for each successive trial in the Data Table. A normality calculation will be made for each trial, and the average of these will be considered to be the normality of the vinegar.

CALCULATIONS: 1. *Normality of vinegar.* The volumes of vinegar and sodium hydroxide solutions used in each trial and the normality of the standard hydroxide solution are known. Solve the *inverse* proportion (see Introduction section of Experiment 28) to find the normality of the vinegar for each trial. Determine the average *value* of the normalities from the different trials. Record each result in the Data Table.

2. *Equivalents of acetic acid in 100 ml of vinegar.* The formula of acetic acid may be

DATA TABLE

Student answers.

	First Trial	Second Trial	Third Trial
Initial reading, acid buret	ml	ml	ml
Final reading, acid buret	ml	ml	ml
Volume of acid used	ml	ml	ml
Initial reading, hydroxide buret	ml	ml	ml
Final reading, hydroxide buret	ml	ml	ml
Volume of hydroxide used	ml	ml	ml
Normality of standard hydroxide	*N*	*N*	*N*
Normality of vinegar (computed)	*N*	*N*	*N*
	Average value		*N*
Percentage of acetic acid in vinegar			%

written as $HC_2H_3O_2$ to show that it is a *monoprotic* acid. What is the g-eq. wt. of acetic acid? A normal solution, by definition, contains the g-eq. wt. (1 equivalent) of solute per liter of solution. Thus,

$$\text{normality} = \frac{\text{no. equivalents solute}}{\text{no. liters solution}}$$

and

$$\text{no. equivs solute} = \text{normality} \times \text{no. liters sol'n}$$

Knowing the normality (average value) of the vinegar, determine the number of equivalents of $HC_2H_3O_2$ in 10$\bar{0}$ ml of the vinegar. Show computations in smooth form on a separate sheet of paper.

3. *Percentage of acetic acid in vinegar.* Find the number of grams of acetic acid in 10$\bar{0}$ ml of vinegar. This is determined from the number of equivalents of acetic acid per 10$\bar{0}$ ml of vinegar and the number of grams of acetic acid per equivalent (g.-eq. wt.):

$$\text{no. g } HC_2H_3O_2/100\text{-ml vinegar} = \frac{\text{no. equivs}}{10\bar{0}\text{ ml}} \times \frac{\text{g } HC_2H_3O_2}{\text{equiv}}$$

The mass percentage of solute in a solution may be expressed on the basis of grams of solute per 10$\bar{0}$ grams of solution. By neglecting the slight difference in density between water and vinegar, we may assume 10$\bar{0}$ ml of vinegar to weigh 10$\bar{0}$ g. Thus, when the mass of acetic acid in 10$\bar{0}$ grams of vinegar is known, the mass-percentage composition may be expressed:

$$\%\ HC_2H_3O_2 = \frac{\text{g } HC_2H_3O_2}{10\bar{0}\text{ g vinegar}} \times 100\%$$

Show your computations in smooth form and record the percentage of acetic acid in vinegar in the Data Table.

4. *(Optional).* This experiment may also be carried out by using molarities. The standard sodium hydroxide solution is approximately 0.5-*M*. The empirical equation is

$$HC_2H_3O_2 + NaOH \rightarrow H_2O + NaC_2H_3O_2$$

The number of moles of sodium hydroxide is calculated from its known molarity and the volume used. Thus

$$\text{molarity} = \frac{\text{no. moles solute}}{\text{no. liters solution}}$$

and

$$\text{moles solute} = \text{molarity} \times \text{no. liters sol'n}$$

The balanced equation shows that 1 *mole* of NaOH is used for 1 *mole* of $HC_2H_3O_2$; NaOH and $HC_2H_3O_2$ are chemically equivalent, mole for mole. Therefore the number of moles of $HC_2H_3O_2$ used in the titration is equal to the number of moles of sodium hydroxide. Calculate the molarity (average value) of the vinegar. Calculate the number of moles of $HC_2H_3O_2$ in 10$\bar{0}$ ml of the vinegar. Determine the number of grams of acetic acid in 10$\bar{0}$ ml of vinegar:

$$\text{no. g } HC_2H_3O_2/10\bar{0}\text{ ml vinegar} = \frac{\text{no. moles}}{10\bar{0}\text{ ml}} \times \frac{\text{g } HC_2H_3O_2}{\text{mole}}$$

The mass percentage of solute in a solution may be expressed on the basis of grams of solute per 100 grams of solution. By neglecting the slight difference in density between water and vinegar, we may assume 10$\bar{0}$ ml of vinegar to weigh 10$\bar{0}$ g. Thus when the mass of acetic acid in 10$\bar{0}$ grams of vinegar is known, the mass-percentage composition may be expressed:

$$5\ HC_2H_3O_2 = \frac{\text{g } HC_2H_3O_2}{10\bar{0}\text{ g vinegar}} \times 100\%$$

Record the percentage of acetic acid in vinegar in the Data Table.

Experiment 30

NAME . . . . . PERIOD . . . . . DATE . . . . . .

INSTRUCTOR'S COMMENTS . . . . . .

Adsorption

PURPOSE: To determine the amounts of dissolved substance adsorbed at different solution concentrations.

APPARATUS: Balance, sensitive to 0.01 g; beakers; 2 burets, 50 ml; clamp, double buret; 4 corks; 5 flasks, Erlenmeyer; flask, volumetric, 250 ml; funnel; graduated cylinder; medicine dropper; pipet, 10 ml; ring, iron; ring stand; wash bottle.

ADDITIONAL APPARATUS for INSTRUCTOR'S EXPERIMENT: ammonia generator (see Exp. 57); beaker, 100 ml; burner and tubing; cork; flask, Erlenmeyer; funnel; ring, iron; ring stand; wire gauze.

MATERIAL: Charcoal, activated; filter paper; standard solutions of acetic acid (approximately 0.4 *M*), sodium hydroxide (approximately 0.1 *M*); succinic or benzoic acid, A. R. grade.

ADDITIONAL MATERIAL for INSTRUCTOR'S EXPERIMENT: ammonium chloride; calcium hydroxide; charcoal, activated. litmus paper, pink; solutions of copper(II) sulfate (1 *M*); methylene blue (1 g in 20 ml water).

INTRODUCTION: Adsorption is defined as the concentration of a gas, liquid, or solid on the surface of a solid or liquid. The substance adsorbed is known as the *adsorbate*, and the substance upon whose surface adsorption occurs is known as the *adsorbent*. Adsorption is selective. A given *adsorbent* will preferentially adsorb more of one substance than of another present in the same environment.

In this experiment, we shall prepare several solutions of acetic acid having equal volumes but different concentrations. Equal quantities of activated charcoal will be introduced into each solution. The amount of acetic acid adsorbed on the charcoal in each solution may then be determined by titrating each against a standard solution of sodium hydroxide.

As the molarity of the standard acetic acid solution from which the test solutions are made is known, and the number of milliliters in each test solution is known, the quantity of acetic acid in each test solution may be expressed in *millimoles* of $HC_2H_3O_2$. Similarly, after adsorption, the quantity of NaOH required to neutralize the remaining $HC_2H_3O_2$ may be expressed in *millimoles* of NaOH. The millimoles of NaOH used in each titration is, of course, equal to the millimoles of $HC_2H_3O_2$ remaining in the test solution. These computations are made as follows:

1. ml of $HC_2H_3O_2$ × Molarity of $HC_2H_3O_2$ = millimoles of $HC_2H_3O_2$ in sample.
2. ml of NaOH × Molarity of NaOH = millimoles of NaOH used.
3. Millimoles of NaOH used = millimoles of $HC_2H_3O_2$ remaining in sample.
4. Millimoles of $HC_2H_3O_2$ — millimoles of $HC_2H_3O_2$ remaining = millimoles of $HC_2H_3O_2$ adsorbed.

SUGGESTION: If two laboratory periods are available for this experiment, the instructor may supply the standard NaOH solution and allow each student to prepare his own $HC_2H_3O_2$ solution and standardize it by titrating against the NaOH solution, using phenolphthalein as an indicator. The first laboratory period should be devoted to the preparation of the test solutions and the addition of the activated charcoal to each.

INSTRUCTOR EXPERIMENT: Add 5 g of activated charcoal to 20 ml of methylene blue solution in a 100-ml beaker. Stir, heat gently, and filter. Repeat with solution of copper(II) sulfate. What does this show about the nature of adsorption?

Add 5 g of activated charcoal to an Erlenmeyer flask previously filled with ammonia. Stopper and shake the flask vigorously. Test for the odor of ammonia. Try moist red litmus paper. Remove the stopper and heat the flask. Repeat the odor and litmus tests. What does this show about adsorption?

STANDARD SOLUTIONS: 1. *NaOH solution.* Prepare an approximately 0.1-*M* NaOH solution by dissolving about 4 g of A.R. grade NaOH pellets in distilled water and dilute to one liter. Standardize with A.R. grade succinic or benzoic acid (0.2–0.3-g samples), using phenolphthalein as the indicator. Determine the molarity and label the flask accordingly.

2. *$HC_2H_3O_2$ solution* (Student quantity). Prepare an approximately 0.4-*M* $HC_2H_3O_2$ solution by dissolving about 6 ml of glacial acetic acid in distilled water and diluting to

250 ml (mix thoroughly). Standardize by titrating 10-ml samples against the standard sodium hydroxide solution, using phenolphthalein. Calculate the molarity and record in the Data Table.

PROCEDURE: 1. All apparatus must be thoroughly clean and dry. Fit each of four 125-ml Erlenmeyer flasks with a cork stopper and number in sequence. Label the $HC_2H_3O_2$ buret to avoid confusion and rinse it with a few milliliters of the $HC_2H_3O_2$ solution. Place it in the clamp, fill, and adjust the level on the scale.

Run 40.0 ml of the $HC_2H_3O_2$ solution into the No. 4 flask. Refill and adjust the buret and then run 20.0 ml into the No. 3 flask, 10.0 ml into No. 2, and 5.0 ml into No. 1. Add 20.0 ml of distilled water to the No. 3 flask, 30.0 ml of distilled water to the No. 2 flask, and 35.0 ml to the No. 1 flask.

2. Weigh out to the nearest 0.01 g four 5-g samples of activated charcoal and add one sample to each of the four flasks. Record the precise mass of each sample in the appropriate place in the Data Table. Shake the four flasks for about 10 minutes. If two laboratory periods are to be used, the stoppered flasks should be set aside and the experiment completed the next day.

3. Set up the NaOH buret as in Part 1 and adjust for titrating. Using a 10-ml pipet, withdraw 20.0 ml of the clear solution from No. 4 flask (filter if necessary) and transfer to a clean Erlenmeyer flask. Add 25 ml of distilled water to provide volume and titrate with the standard NaOH solution, using phenolphthalein as the indicator. Multiply the milliliters of NaOH solution used by 2 (Why?) and record in the Data Table. In a similar manner, titrate a 20.0-ml sample of each of the remaining test solutions. Remember to record twice the number of milliliters of NaOH solution used in each titration. Make the necessary computations on a separate sheet of paper. Complete the Data Table, referring to the Introduction as necessary.

4. (*Optional*). Construct a graph, plotting *millimoles of $HC_2H_3O_2$ adsorbed per gram of charcoal* as ordinate against *millimoles of $HC_2H_3O_2$ remaining* as abscissa. This is a *smooth curve* and ideally gives a sweeping parabola. Ask your instructor for suggestions in regard to the proper construction of your graph.

QUESTIONS: 1. What relationship exists between the concentration of adsorbate and the amount of adsorption? The greater the concentration of the adsorbate, the greater is the amount of adsorption.

2. Recognizing that the molecular concentration of water was greater than that of acetic acid in each test solution, how do you account for the adsorption of acetic acid? Adsorption is specific and depends on the nature of the material, acetic acid, which is being adsorbed.

DATA TABLE

Student answers.

Molarity of standard NaOH solution ..... *M*

Molarity of standard $HC_2H_3O_2$ solution ..... *M*

Volume of each test solution ..... ml

Test solution	ml $HC_2H_3O_2$ added	millimoles $HC_2H_3O_2$ present	g charcoal added	ml NaOH required	millimoles $HC_2H_3O_2$ remaining	millimoles $HC_2H_3O_2$ adsorbed	millimoles $HC_2H_3O_2$ adsorbed per g charcoal
1							
2							
3							
4							

Experiment 31

NAME . . . . . PERIOD . . . . . DATE . . . . . .

INSTRUCTOR'S COMMENTS . . . . . .

Carbon

PURPOSE: To study the occurrence, methods of preparation, and some of the properties of the various forms of carbon.

APPARATUS: Balance, platform; beaker, 250 ml; burner and tubing; clamp, buret; clamp, test tube; crucible; crucible cover; flask, Erlenmeyer; forceps; funnel; graduated cylinder; molecular model kit (Sargent); rubber stoppers, sold No. 2 (3), solid No. 4; ring, iron; ring stand; test tubes; triangle, pipestem.

MATERIAL: Alcohol, denatured; boneblack, powd.; charcoal, activated, pellets; charcoal, wood, powd.; charcoal, wood, splinters; copper(II) oxide, powd.; detergent, granular; graphite, powd.; lampblack; sugar; filter papers; splints, wooden; hydrochloric acid, dilute (1:4); nitric acid, dilute (1:5); solutions of dark-brown sugar; hydrogen sulfide (very dilute); lead acetate (1.0 *M*); limewater; sodium hydroxide (1.0 *M*). Instructions for preparing the special dark-brown sugar solution and the very dilute hydrogen sulfide solution are given in TEACHERS' EDITION TO EXERCISES AND EXPERIMENTS IN CHEMISTRY.

INTRODUCTION: Carbon exhibits allotropy, the existence of an element in two or more forms in the same physical state. Its two crystalline allotropic forms are diamond and graphite. Black residues obtained by heating substances which contain combined carbon are sometimes called amorphous carbon. Examples of amorphous carbon include charcoal, coke, boneblack, and lampblack. Carbon atoms form covalent bonds with other elements and link with each other in different ways to produce the diamond and graphite structures. The diamond structure is the result of sp^3 hybridization while the resonant graphite structure probably involves a different type of hybridization known as sp^2. The amorphous forms are produced by a variety of procedures including destructive distillation and incomplete combustion. Carbon is an important reducing agent. The uses of its different varieties are related to particular properties including combustibility and adsorption.

PROCEDURE: 1. Each student team is to use two molecular model sets, each of which contains 20 black carbon spheres. Using the short stick connectors, construct a model of the diamond. See Fig. 31-1. Start the construction of the model of graphite by constructing two hexagons using only the short connectors. Recall that the distances between the centers of adjacent carbon atoms in a layer of graphite are identical, 1.42 Å. Using Fig. 31-2 as a model, connect the two hexagons, using the longer stick connectors to represent the distance between centers in adjacent layers (3.40 Å). Use the remaining carbon spheres to build up each layer. Two carbons in the existent hexagons (those first constructed) are to be common to the newly constructed hexagons. Use long connectors, as needed, between the rest of the constructed layers. To how many carbons in a layer in graphite is each carbon bonded? What is the nature of the bonding between layers in graphite?

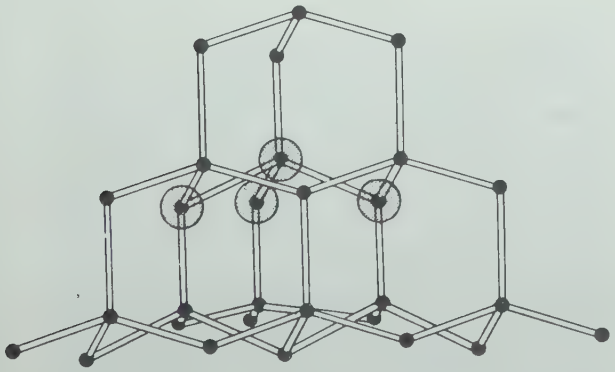


Fig. 31-1

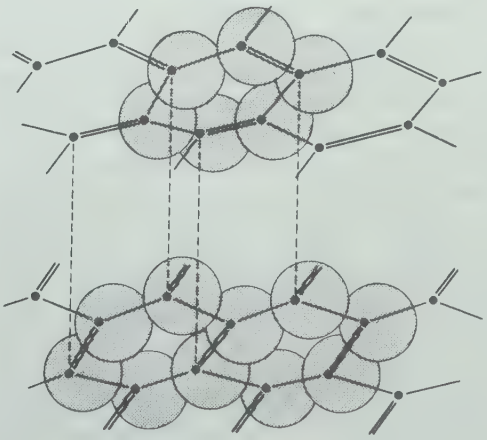


Fig. 31-2

Observations: Each carbon is bonded to 3 carbons in the hexagon layer. The layers are held together by weak dispersion interaction forces.

2. Ignite a wood splint in the burner flame. Note how it burns. Then break several splints and place the pieces in the bottom of a test tube, clamped so its mouth is slightly downward. Place a piece of paper on the table under the mouth of this test tube and heat the contents strongly (until no more volatile matter is released). **CAUTION:** *Do not breathe the vapor or get any of the liquid on your hands.* Remove the residue from the test tube carefully. Describe its appearance. Ignite one piece in the burner and observe how it burns. Pour one inch of limewater into a small test tube. Ignite another piece of the solid residue (hold with forceps) and insert the ignited end in the test tube, holding it above the limewater. If nothing happens to the limewater, repeat with another ignited piece. What happens to the limewater? What does this indicate about the nature of the residue? What name is given to the process employed in heating the wood in the test tube?

Devise and conduct an experiment to show that sugar may be decomposed into carbon and water vapor. Use 2 g of sugar in a porcelain crucible set in a pipestem triangle.

Hold a test tube horizontally with its closed end in the *yellow* burner flame. What form of carbon is produced? Change to the standard blue flame and hold the test tube so that the deposit is in the hottest part of the flame. Note the results.

Observations: Carbon black is deposited on the test tube. The deposit is removed by the oxidizing blue flame.

3. Put 15 ml of water in each of three test tubes. Into the first, add 2 g of powdered graphite. Into the second, add 2 g of powdered wood charcoal. Into the third, add 2 g of lampblack. Shake each sample vigorously and then pour out half the contents of each tube into a second set of test tubes. Place the first set of tubes in the test tube rack and look for signs of solution or suspension of carbon. Into each of the second set place 3 grains of a solid detergent such as Naccanol. Stopper and shake each test tube. After 5 minutes record any instance of settling. Reshake these samples and filter each. Which mixtures pass through the filter paper? In what condition was the carbon when shaken with water? When mixed with a detergent (wetting agent)? Which carbon sample is apparently in the smallest state of subdivision?

Observations: Student answer.

4. Devise and conduct experiments to determine the activity and solubility of wood charcoal splinters in dilute hydrochloric acid, dilute nitric acid, water, alcohol, and sodium hydroxide solution. Describe your experiments and indicate their results. Discard the liquids into the sink, but be sure that any pieces of charcoal which remain are placed in the waste jar and *not in the sink.*

Observations: Student answer.

5. Pour 50 ml of dark-brown sugar solution into an Erlenmeyer flask. Add to it 1 g of powdered boneblack and stopper the flask. Shake the mixture vigorously for a minute. Filter. Pour the filtrate through the funnel a second time, and even a third time if necessary to remove the color from the sugar solution. Finally, compare the color of the filtrate with that of the original brown-sugar solution.

Observations: Student answer.

6. Fill a test tube one-half full of very dilute hydrogen sulfide solution. Cautiously note its odor using the method shown in Fig. F-5. **CAUTION:** *Hydrogen sulfide is very poisonous.* As a chemical test for its presence add 3 drops of the solution to 2 ml of a solution of lead acetate. Result? Add 5 pellets of activated charcoal to the test tube and shake vigorously at intervals for several minutes. Decant and note the odor of the decantage. Drop 3 drops of the decantate into 2 ml of lead acetate solution. Result?

Observations: Student answer.

7. Mix 5 g of powdered copper(II) oxide with an equal bulk of powdered wood charcoal. Heat this mixture strongly for 4 or 5 minutes in a test tube. You should observe a reaction occurring in the test tube during this heating. After the test tube cools, pour its contents into a beaker. Devise a method of washing the excess low density charcoal away from the much higher density reaction product. Identify the reaction product.

Observations: Student answer.

QUESTIONS: 1. How active an element is carbon at room temperature?

Carbon is inactive at room temperature.

2. Of what use is charcoal in water purification processes? To remove objectionable odors from the water.

3. What is the nature of the reaction between hot copper(II) oxide and carbon?

This is an oxidation-reduction reaction. The carbon reduces the copper in the copper(II) oxide to elementary copper. The carbon is oxidized to carbon dioxide.

4. What would you predict is the reaction between hot iron(III) oxide and carbon?

The carbon would reduce the iron in iron(III) oxide to elementary iron. The carbon would be oxidized to carbon dioxide.

5. Carry out the reaction in Question 4 to verify your prediction. Result? Student answer.

6. What is adsorption? Why are certain forms of charcoal good adsorbing agents?

Adsorption is the concentration of a gas, liquid, or solid on the surface of a liquid or solid with which it is in contact. Charcoal has tremendous surface of particles of colloidal dimensions. Charcoal may be activated by treating it with steam.

EQUATIONS

1. Reaction between hydrogen sulfide and lead acetate $H_2S + Pb(C_2H_3O_2)_2 \rightarrow PbS(s) + 2\ HC_2H_3O_2$.
2. Reaction in Part 7 $2\ CuO + C \rightarrow 2\ Cu + CO_2(g)$.

Experiment 32

NAME . . . . . PERIOD . . . . . DATE . . . . . .

INSTRUCTOR'S COMMENTS . . . . . .

Carbon Dioxide

PURPOSE: To prepare carbon dioxide by a variety of methods and to study its properties.

APPARATUS: Balance, platform; beaker, 250 ml; bottles, wide mouth; burner and tubing; clamp, buret; flask, Erlenmeyer; forceps; funnel tube; glass bends, right angle (2); glass bend, right angle, long arm; glass plates; graduated cylinder; ring stand; rubber stoppers, 1-hole No. 2, 2-hole No. 6; rubber tubing, small diameter; test tubes; trough; wing top.

ADDITIONAL APPARATUS for INSTRUCTOR'S EXPERIMENT: bottle, gallon with delivery tube assembly; bottle, wide mouth.

MATERIAL: Copper(II) carbonate; magnesium ribbon; marble chips; sodium aluminum sulfate; sodium carbonate, anhydrous; sodium hydrogen carbonate; candle; wooden splints; hydrochloric acid, conc.; hydrochloric acid, dilute (1:4); sulfuric acid, dilute (1:6); limewater; litmus solution.

ADDITIONAL MATERIAL for TEACHER'S EXPERIMENT; mineral oil; molasses; yeast; solution of limewater.

INTRODUCTION: Carbon dioxide may be prepared by a variety of methods including heating certain carbonates, combustion of ordinary fuels, and fermentation of sugar. The usual laboratory method consists of adding an acid, such as hydrochloric acid, to a carbonate. Carbonic acid, H_2CO_3, is first produced, but it is unstable and decomposes readily into carbon dioxide and water. Carbon dioxide is moderately soluble in water, but it can be collected by water displacement if the gas is evolved rather rapidly.

PROCEDURE: 1. Thrust a blazing wooden splint into a bottle and keep it there until it is extinguished. Pour limewater to a depth of about 1 cm into the bottle, cover with a glass plate and shake. Result?

Put 2 grams of copper(II) carbonate in a test tube. Insert a one-hole rubber stopper with a long-arm glass-bend delivery tube in the mouth of the test tube. Clamp to a ring stand. Pour limewater to a depth of 2.5 cm into a second test tube and insert the long arm of the delivery tube in the limewater. Heat the test tube containing the copper(II) carbonate vigorously until you see a permanent change in color. What happens to the limewater? Repeat this procedure first with a sample of anhydrous solid sodium carbonate (representing the carbonates of Group I) and then with a sample of sodium hydrogen carbonate. Results?

Repeat Part 5, Experiment 18, which deals with the addition of water to the typical ingredients of a baking powder. Result?

Put about 1 g of sodium hydrogen carbonate, 1 g of sodium hydrogen carbonate, and several marble chips in separate test tubes. To each add 3 ml of dilute hydrochloric acid. Results? Into the upper part of each test tube thrust a blazing splint when the reactions have subsided. Results? Repeat this procedure with separate solid samples, using dilute sulfuric acid instead of hydrochloric acid. Results? What conclusions do you reach?

INSTRUCTOR EXPERIMENT: Mix a cake of compressed yeast or a packet of dried yeast with 100 ml of warm water to form a milky suspension. Add this to a fallon bottle containing 500 ml of molasses diluted with warm water to make 3 liters of solution. Insert a delivery tube assembly in the mouth of the bottle and have the delivery end extend well into a wide-mouth bottle containing limewater over which a thin film of mineral oil has been poured. Put in a warm place and after a day have students observe the action in the limewater bottle. What fermentation product does this indicate?

Observations: Limewater turns milky due to precipitation of $CaCO_3$ indicating production of CO_2.

2. Set up an apparatus for generating a gas by the action of a solution on a solid and for collecting the gas by water displacement. Put 50 g of marble chips in the generator and cover it with water. Why? Using an appropriate technique, add concentrated hydrochloric acid, a little at a time, to produce a constant evolution of gas. Collect two bottles of gas. What physical properties of carbon dioxide do you observe?

Observations: The generator flask contains a funnel tube, the bottom of which must be covered by a liquid in order to prevent escape of CO_2 through the funnel tube. Water addition also dilutes the concentrated acid so that

the reaction is not too vigorous. Collection by water displacement indicates that the gas is slightly soluble. The gas is colorless.

3. Into one bottle of carbon dioxide, thrust a blazing wooden splint. Then, with forceps, thrust a 5-cm piece of burning magnesium ribbon into the same bottle. Are the reactions similar? Explain. Note the two different solid reaction products formed from magnesium and carbon dioxide.

Observations: The blazing splint goes out while the burning Mg continues to burn vigorously forming a black deposit of carbon and a white deposit of MgO. The burning Mg is hot enough to decompose CO_2.

4. Place a short piece of candle, not over 2 cm long, in a 250-ml beaker and light it. Rapidly pour the carbon dioxide from the second bottle you collected into the beaker, just as if you were pouring water. What physical and chemical properties of carbon dioxide are demonstrated?

Observations: CO_2 is heavier than air and does not support combustion.

5. Bubble carbon dioxide from the generator through a test tube one-half full of limewater. Observe the results. This is a test for carbon dioxide.

Observations: Student answer.

6. Using a glass tube, blow your breath through a test tube one-third full of limewater for at least 2 minutes. Result? What conclusion can you draw from this experiment?

Observations: Student answer.

7. Bubble carbon dioxide from the generator for a few minutes through a test tube one-third full of neutral litmus solution. Result? Why?

Observations: The litmus turns red because of the formation of weak carbonic acid.

QUESTIONS: 1. Carbon dioxide may be collected by water displacement or by upward displacement of air. Give an advantage and disadvantage of each method.

Water displacement advantage: carbon dioxide is fairly pure; disadvantage: carbon dioxide is moderately soluble in water. Air displacement advantage: less apparatus is involved; disadvantage: carbon dioxide is mixed with air.

2. In Part 3, what evidence is there that the carbon dioxide was decomposed?

A black deposit of carbon collects on the walls of the bottle.

3. What use for carbon dioxide is suggested in Part 4? As a fire extinguisher.

FURTHER EXPERIMENTATION: Using the Sargent molecular model kit, construct a model of one of the resonance hybrids of carbon dioxide.

Bubble carbon dioxide through limewater until the precipitate formed redissolves. Heat a sample of this solution to boiling.

Heat a marble chip on the grid of a Fisher burner for 15 minutes. Permit the residue to cool. Put it in a small beaker and add 5 ml of water. Test the material produced with red and blue litmus papers.

Perform the limewater test on the gas given off by carbonated beverages.

EQUATIONS

1. Complete combustion of carbon. $C + O_2 \rightarrow CO_2(g)$.
2. Decomposition of copper(II) carbonate (heat). $CuCO_3 \rightarrow CuO + CO_2(g)$.
3. Decomposition of sodium hydrogen carbonate (heat). $2NaHCO_3 \rightarrow Na_2CO_3 + H_2O(g) + CO_2(g)$.
4. Preparation of carbon dioxide (laboratory method). $CaCO_3 + 2HCl \rightarrow CaCl_2 + H_2O + CO_2(g)$.
5. Fermentation of glucose ($C_6H_{12}O_6$) (molasses). $C_6H_{12}O_6 \rightarrow 2C_2H_5OH + 2CO_2(g)$.
6. Carbon dioxide and burning magnesium. $CO_2 + 2Mg \rightarrow 2MgO + C$.
7. Reaction in Part 5. $CO_2 + Ca(OH)_2 \rightarrow CaCO_3(s) + H_2O$.
8. Carbon dioxide and water. $H_2O + CO_2 \leftrightarrows H_2CO_3$.
9. Two equations representing the successive steps in the ionization of carbonic acid in water. $H_2CO_3 + H_2O \rightleftarrows H_3O^- + HCO_3^-$.

$HCO_3^- + H_2O \rightleftarrows H_3O^+ + CO_3^{--}$.

Experiment 33

NAME . . . . . PERIOD . . . . . DATE . . . . . .

INSTRUCTOR'S COMMENTS . . . . . .

Hydrocarbons

PURPOSE: To construct models of representative members of various hydrocarbon series. To prepare and collect methane and ethyne and to study some of their properties.

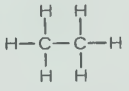
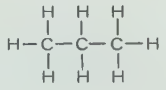
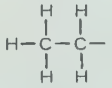
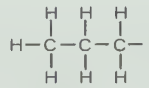
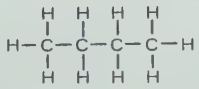
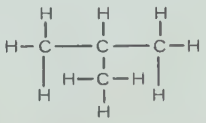
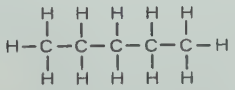
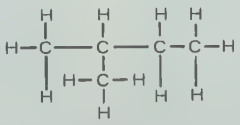
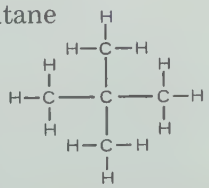
APPARATUS: Balance, platform: bottles, wide-mouth (5); burner and tubing; clamp, buret; flask, Florence; funnel; glass bends (2); glass plates (4); glass tube; graduated cylinder; molecular model kit, Sargent; mortar and pestle; pinch clamp; ring, iron; ring stand; rubber connector; rubber stopper, solid, No. 2; rubber stopper, 1-hole No. 4; rubber stopper, 2-hole No. 6; test tube, large, Pyrex; test tubes; trough; tubing, delivery; wing top.

MATERIAL: calcium carbide, lump; soda lime; sodium acetate, anhydrous, powdered; solutions of bromine water; bromine in carbon tetrachloride; limewater; splints, wooden.

INTRODUCTION: The hydrocarbons exist in various series. Their structural formulas may be better visualized through the constructions of ball-and-stick models. The shapes of many of the hydrocarbons are accurately reproduced by such models although the role of various *p* orbitals and hybridized orbitals is not necessarily evident. The saturated hydrocarbons react slowly with reagents such as bromine (water solution or carbon tetrachloride solution) by the process of substitution. The unsaturated hydrocarbons react much more rapidly by the process of addition. Practically all hydrocarbons will burn more-or-less completely in air.

SUGGESTION: Review Chapters 6 and 17 of MODERN CHEMISTRY, 1974, particularly those parts dealing with hybridized orbitals and the role of the *p* orbitals in determining the shapes of molecules. Perchloroethylene (C_2Cl_4) may be used instead of CCl_4.

DATA TABLE 1

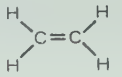
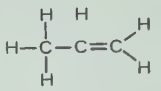
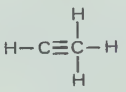
Alkanes methane CH_4	ethane C_2H_6 	propane C_3H_8 
methyl group CH_3^-	ethyl group $C_2H_5^-$ 	propyl group $C_3H_7^-$ 
n-butane C_4H_{10} 	isobutane C_4H_{10}  other name 2-methylpropane	
n-pentane C_5H_{12} 	isopentane C_5H_{12}  other name 2-methylbutane	neopentane C_5H_{12}  other name 2, 2-dimethylpropane

PROCEDURE: 1. General directions: Assemble ball and stick models of the various hydrocarbon series using the Sargent molecular model kit. The short rods are used to connect the hydrogen spheres to the carbon spheres. The long rods are used to connect carbon spheres to each other. The springs are used for multiple bonds between carbon spheres. Before starting, check your kit for completeness using the color code attached to the inside of the box cover. After a number of models have been constructed, and their conformity with structural formulas checked by writing the structural formulas, they will have to be disassembled so that the next successive models may be constructed. Complete Data Table 1. Under each name write the molecular formula. In the space at the right place the structural formula.

By what group do the formulas for successive alkanes differ from the preceding one? Identify the types of bonds in ethane: the carbon-carbon bond, and the carbon-hydrogen bonds.

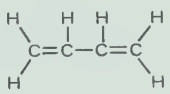
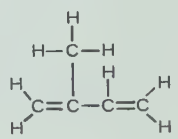
Observations: Alkanes successively differ by a CH_2- group. In ethane the C–C bond is an sp^3-sp^3 bond while the carbon-hydrogen bonds are sp^3-s bonds.

DATA TABLE 2

Alkenes ethene C_2H_4  other name . . . . ethylene	propene C_3H_6 
Alkynes ethyne C_2H_2 $H-C\equiv C-H$ other name . . . . acetylene	C_3H_4 propyne 

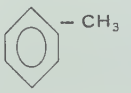
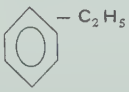
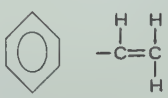
By what group do the formulas for the successive alkenes differ from the preceding one? What is the shape of the ethene molecule? Of the ethyne molecule? What two types of bonds constitute the double bond in ethyne? What types of bonds constitute the triple bond in ethyne? What is the shape of the two $2p$ orbitals which overlap? How do the carbon-to-carbon distances vary in the series: ethane, ethene, ethyne? Complete Data Tables 2 and 3.

DATA TABLE 3

Alkadienes 1,3 butadiene C_4H_6 	isoprene (2-methyl-1,3-butadiene) C_5H_8 

DATA TABLE 4

Aromatic: Construct the more complex models on the benzene structure.

benzene C_6H_6	toluene C_7H_8  $-CH_3$ other name . . . . methyl benzene	phenyl group C_6H_5-
ethyl benzene  $-C_2H_5$ C_8H_{10}	naphthalene $C_{10}H_8$	styrene  C_8H_8

Observations: Successive alkenes differ by a CH_2-group. Ethene molecule is planar. Ethyne molecule is linear. In ethene, the double bond consists of an sp^2-sp^2 bond and a *p*-*p* overlap bond. The triple bond in ethyne consists of an *sp*-*sp* and two p-p overlap bonds. The two overlapping *p* orbitals are at right angles to each other. The carbon-carbon distances are: for ethane—1.53A; for ethene—1.33Å, for ethyne—1.20Å.

What is the shape of the benzene molecule? What is believed to be the nature of the carbon-carbon bonding in benzene? What is the size of the carbon-hydrogen bond angles in benzene? Briefly describe the role of the *p* orbitals in each carbon atom in the benzene structure. Complete Data Table 4.

Observations: The benzene molecule is hexagonal planar. The carbon-carbon bonding is believed to be sp^2-sp^2. The carbon-hydrogen bond angles are 120°. The *p* orbitals above and below the molecular plane overlap to form three orbitals, the principle one of which consists of two doughnut-shaped rings one above and one below the plane of the molecule.

2. *Methane.* Grind 8 g of powdered sodium acetate with 14 g of soda lime in a mortar. Add the mixture to a large Pyrex test tube and incorporate the test tube in an apparatus for generating a gas by heating a mixture of solids and collecting the gas by water displacement. Distribute the mixture evenly at the back of the test tube with an air space above. Why? Heat the test tube, gently at first, and then more strongly. Collect one-half bottle of gas and discard it. Why? Then collect one full test tube of methane and stopper it. Collect two full bottles of methane and complete the filling with methane of a bottle 9/10 full of air and 1/10 full of water. Remove the end of the delivery tube from the water in the trough. Remove the flame from under the generator. What physical properties of methane have you observed? **CAUTION:** *Do not collect methane or ethyne in any bottle that holds more than 250 ml.* Why?

Observations: Solid mixture is distributed to insure uniform heating. The first sample contains air as an impurity. Methane is a colorless gas which is practically insoluble in water. Ignition of containers of more than 250 ml may result in violent combustion or explosion.

Ignite a bottle full of methane. Note the color of the flame. Observe the appearance of the walls of the bottle while the methane is burning. Test for the other product of combustion by pouring 5 ml of limewater into the wide-mouth bottle, covering it with a glass plate, and shaking the bottle. Result?

Observations: Methane burns with a blue flame depositing water droplets on the side of the bottle. The other product is CO_2 identified by its reaction with limewater which turns milky.

Place the bottle containing 1/10 methane and 9/10 air mouth upward on the laboratory table. Remove the glass plate and *cautiously* at arm's length bring a lighted splint to the mouth of the bottle. Result? Why does this mixture of methane and air produce such a result?

Observations: A violent explosion rather than the steady burning of methane alone, results. The methane-air mixture is an explosive mixture.

To the methane in another wide-mouth bottle, add 5 ml of bromine water, cover with a glass plate, and shake without inverting the bottle. Result?

Observations: No observable change occurs since methane is a saturated hydrocarbon.

To the methane in the test tube add 1 ml of bromine in CCl_4 and quickly restopper the test tube. Shake the contents and observe the color change, if any, of the bromine solution.

Observations: No color change occurs.

3. *Ethyne.* Add 8 g of calcium carbide lumps to a *dry* Florence flask fitted with funnel, rubber connector, pinch clamp, glass tube, glass bend, stopper, and delivery tub-

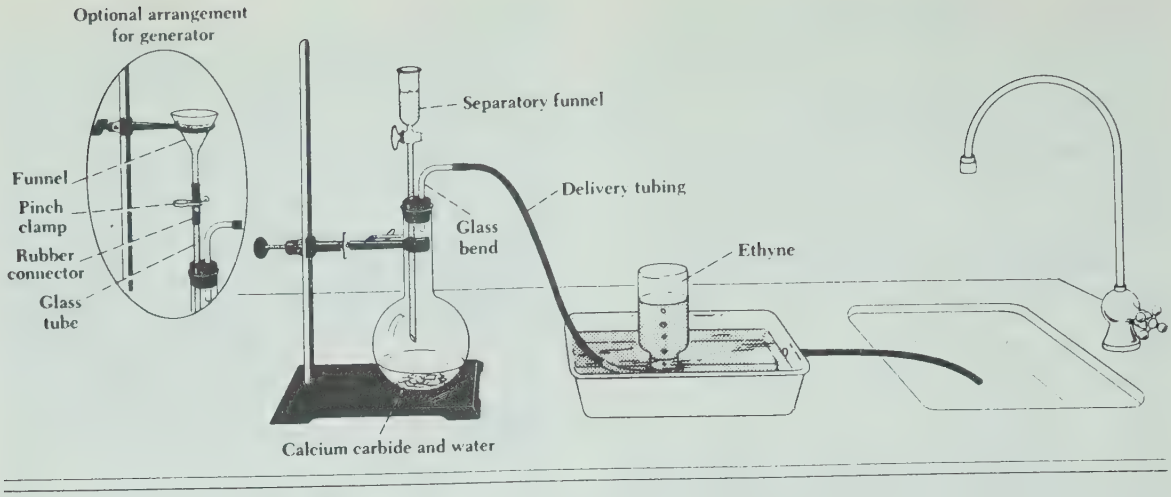


Fig. 33-1

ing as shown in Fig. 33-1. *Have your instructor approve the apparatus. Wrap a towel around the generator.* With the pinch clamp closed, nearly fill the funnel with water. By opening the pinch clamp slightly, allow the water to flow dropwise on the calcium carbide. When vigorous action starts, close the pinch clamp. Collect a bottle of gas by water displacement and discard it. If action in the generator slows, open the pinch clamp to admit more water. Then collect three bottles of the gas. Finally complete the filling with gas of a bottle which contains 14/15 air and 1/15 water. **CAUTION:** *Before lighting your burner*, dismantle the generator under the hood, and dispose of calcium carbide as directed by your instructor. What physical properties of ethyne have you observed?

Observations: Ethyne is a colorless gas which is practically insoluble in water.

Cautiously at arm's length, bring a lighted splint to the uncovered mouth of the bottle containing the ethyne-air mixture as it rests mouth upward on the laboratory table. Result?

Observations: An explosion results.

Ignite a full bottle of ethyne. Note the color of the flame and one of the products of combustion. Blow hard across the mouth of the bottle. Result? Add 5 ml of limewater to the bottle and shake. Result?

Observations: The flame is yellow and black specks of soot (unburned carbon) float in the air and coat the upper parts of the tube. Blowing hard across the mouth of the bottle may cause the flame to become brighter. Limewater turns milky indicating production of CO_2.

To another bottle of ethyne, add 5 ml of bromine water. Cover with a glass plate and shake without inverting the bottle. Result?

Observations: The bromine color disappears due to the addition of Br_2 to the unsaturated ethyne.

To the last bottle of ethyne add 1 ml of bromine in CCl_4. Cover with glass plate, shake vigorously without inverting the bottle and note the color of the bromine solution.

Observations: The decolorization of bromine solution takes place again because of addition of bromine to the triple bond of ethyne.

QUESTIONS: 1. What is one cause of explosions in coal mines that you have learned from this experiment? An accumulation of methane mixed with the proper proportions of air will explode if ignited.

2. What is necessary in order for ethyne to burn with a clean flame? An abundant supply of air mixes with the ethyne.

3. Hydrogen is odorless. Yet sometimes a peculiar odor is given off when dilute sulfuric acid is added to scrap iron. How can this odor be accounted for? Scrap iron contains iron carbide which reacts with the dilute sulfuric acid to produce a mixture of gaseous hydrocarbons.

4. Name the compounds formed when (a) one bromine atom substitutes for one hydrogen atom in methane, (b) two bromine atoms add to ethene, (c) four bromine atoms add to ethyne. (a) methyl bromide, (b) 1,2-dibromoethane, (c) 1,1,2,2-tetrabromoethane.

FURTHER EXPERIMENTATION: Use your models of methane, ethane, ethene, and ethyne to construct the models of products resulting from the reaction of these hydrocarbons with bromine.

To 3-ml samples of the following liquids in test tubes, add 1 ml of bromine in CCl_4, stopper, and shake vigorously to test for unsaturates; kerosene, gasoline (white), mineral oil, benzene, toluene. Also test a small piece of paraffin and naphthalene in the same manner. Collect a bottle of the fuel gas supplied to the laboratory by water displacement and test it this same way.

Try samples (above) with potassium permanganate solution (0.3%).

Use the molecular model kit to construct the fundamental polymeric units in polyethylene and neoprene.

Cracking: Test 1 ml of mineral oil for the presence of unsaturates using bromine in CCl_4. Heat 2 ml of mineral oil in which a small wad of steel wool has been placed. Remove the steel wool, permit the oil to cool, and test with bromine in CCl_4.

EQUATIONS

1. Preparation of methane $Na_2C_2H_3O_2 + NaOH \rightarrow CH_4(g) + Na_2CO_3$
2. Complete combustion of methane. $CH_4 + 2O_2 \rightarrow CO_2(g) + 2H_2O(g)$
3. Reaction between methane and bromine. $CH_4 + Br_2 \rightarrow CH_3Br + HBr$
4. Preparation of ethyne. $CaC_2 + 2H_2O \rightarrow Ca(OH)_2 + C_2H_2(g)$
5. Complete combustion of ethyne. $2C_2H_2 + 5O_2 \rightarrow 4CO_2(g) + 2H_2O(g)$
6. Reaction between ethyne and bromine. $C_2H_2 + 2Br_2 \rightarrow C_2H_2Br_4$

NAME . . . . . PERIOD . . . . . DATE . . . . . .
INSTRUCTOR'S COMMENTS . . . . . .

Experiment 34 Hydrocarbon Substitution Products

PURPOSE: To construct models of representative hydrocarbon substitution products. To show some of the characteristic reactions of certain hydrocarbon substitution products.

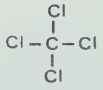
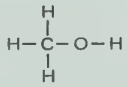
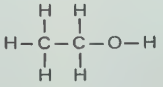
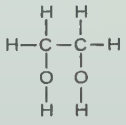
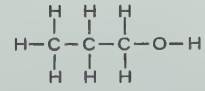
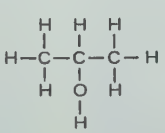
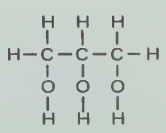
APPARATUS: Balance, platform; burner and tubing; clamp, test tube; cork handle; dish, evaporating; graduated cylinder; medicine dropper; molecular model kit, Sargent; ring, iron; ring stand; test tubes; test tube, large, Pyrex; thermometer; wire gauze.

MATERIAL: Acetic acid, glacial; amyl alcohol; copper wire spiral, 25 cm No. 18 gauge; cottonseed oil; ethanol; glycerol; kerosene; magnesium turnings; methanol; olive oil; salicylic acid; litmus papers, red and blue; ammonia-water solution, dilute (1:4); hydrochloric acid, dilute (1:4); sulfuric acid, conc., solutions of: aniline hydrochloride, sat'd; Fehling's A & B; formaldehyde (40% soln. formalin); silver nitrate (0.1 *M*); sodium hydroxide (10 *M*).

INTRODUCTION: Most of the important classes of organic compounds may be regarded as hydrocarbon substitution products. Their properties, for the most part, depend on the nature of the substitution group. For example, alcohols characteristically contain the hydroxyl group. However their water solutions give no evidence of the hydroxide ion. On the other hand, organic acids, which contain the carboxyl group, generally act as weak acids in water solution. The construction of representative models helps in the study of the nature of the bonding in these substituted groups.

PROCEDURE: 1. *Model construction*, general directions: Assemble ball and stick models of the substances whose formulas are given in Data Tables 1 and 2. Use spring connectors for all types of multiple bonds. Where carbon spheres are joined to other carbons by single bonds, use the long rods. Use the short rods for other single bonds. Before starting, check your kit for completeness and identification using the color code attached to the inside of the box cover. After a number of models have been constructed and their conformity with structural formulas checked, disassemble them so their components may be used with subsequent models. Under each formula write the name of the substance. In the space provided, draw the representative structural formula.

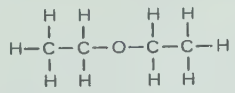
DATA TABLE 1

Alkyl halogenides CH_3Cl methyl chloride	$CHCl_3$ trichloromethane	CCl_4 carbon tetrachloride 
Alcohols CH_3OH methanol 	C_2H_5OH ethanol 	$C_2H_4(OH)_2$ ethylene glycol 
C_3H_7OH propanol 	$CH_3CHOHCH_3$ 2-propanol 	$C_3H_5(OH)_3$ glycerol 

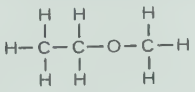
DATA TABLE 2

Ethers

$C_2H_5OC_2H_5$
diethyl ether



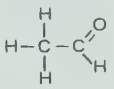
$C_2H_5OCH_3$
methyl ethyl ether



Aldehydes

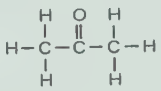
HCHO
formaldehyde

CH_3CHO
acetaldehyde

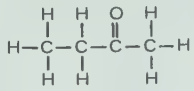


Ketones

CH_3COCH_3
acetone

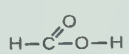


$C_2H_5COCH_3$

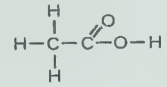


Carboxylic acids

HCOOH
formic (methanoic)

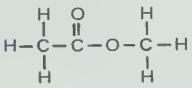


CH_3COOH
acetic (ethanoic)

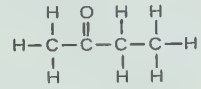


Esters

CH_3COOCH_3
methyl acetate



$CH_3COOC_2H_5$
ethyl acetate



2. *Organic Reactions*

A. Alcohols.

a. Test 5 ml of ethanol in a test tube with red and blue litmus papers. Results? Add 5 ml of water to the ethanol. Shake. Result? Test this mixture with red and blue litmus papers.

b. Repeat with a 5-ml sample of glycerol and then with amyl alcohol. Results?

c. Place 1 ml of methanol in a test tube. Heat a spiral of copper wire to red heat holding it by a cork which serves as a handle. Plunge the heated spiral into the methanol. Repeat several times. Test the odor of the product using the procedure in Fig. F-5. Note the color changes undergone by the spiral when it is heated and then when it is dipped into the methanol. What substance is produced by this oxidation of methanol?

Observations: a & b: No reactions to litmus. Formaldehyde, which has a suffocating odor is produced by the oxidation of the methanol. The heated spiral becomes black with the production of CuO which then turns back to red copper during the oxidation.

B. Carboxylic acids

Test 2 ml of glacial acetic acid in a test tube with red and blue litmus papers. Add 5 ml of water to the acid sample and stir. Repeat the litmus paper tests. Results? Add a few magnesium turnings to 5 ml of dilute hydrochloric acid and a similar amount of turnings to 5 ml of the diluted acetic acid. Results?

Observations: Glacial acetic acid has no effect on litmus. Its water solution turns blue litmus to red. Both acids yield H_2 gas with Mg, the acetic acid reacting much less vigorously.

C. Esters

a. To 5 ml of acetic acid, CH_3COOH, in test tube, add 5 to 6 drops of concentrated sulfuric acid and 3 ml of ethanol, C_2H_5OH. Warm gently and note the odor of the product. Of what fruit flavor does it remind you? Name the product.

Observations: The product, ethyl acetate, has a fruity odor characteristic of apples.

b. Put about 1 g of salicylic acid, $C_6H_4OHCOOH$, in an evaporating dish and add to it 1 ml of methanol, CH_3OH. Next, add 3 drops of concentrated sulfuric acid and warm gently. You should be able to identify the odor. Name the product.

Observations: The product, methyl salicylate, has the odor of oil of wintergreen.

c. To 10 ml of acetic acid in a test tube, add 5 ml of amyl alcohol, $C_5H_{11}OH$, and 2 ml of concentrated sulfuric acid. Warm gently and observe the odor, which is similar to that of a common tropical fruit. Name the product.

Observations: The product amyl acetate has the odor of bananas.

D. Aldehydes.

a. To 5 ml of mixed Fehling's solutions add 3 drops of formalin (formaldehyde solution). Result? Heat to boiling. Result?

b. To 5 ml of silver nitrate solution in a well-cleaned test tube add dilute ammonia-water until a precipitate forms. Then add ammonia-water dropwise with stirring until this precipitate just dissolves. Add 10 drops of formalin and heat the contents until a silver mirror forms. **CAUTION:** The mirror must be destroyed by adding nitric acid since it cannot be safely stored.

Observations: a. Upon heating, the formaldehyde is oxidized and a brick-red precipitate of copper(I) oxide is formed.

E. Aniline ($C_6H_5NH_2$): *Condensation polymerization.*

Pour 10 ml of aniline hydrochloride into a large test tube. Insert a thermometer. Add 10 ml of formalin with stirring. Note the temperature change. Results?

Observations: The temperature rises and a red-brown rather gelatinous solid forms. This hardens and darkens upon standing.

F. Saponification.

a. Mix 4 ml of cottonseed oil or olive oil with 5 ml of ethanol in an evaporating dish. Stir in 3 ml of sodium hydroxide solution and heat over a low flame, stirring constantly, until the mixture has the consistency of mayonnaise. Remove the flame and allow the contents of the evaporating dish to cool. Do you think the reaction would occur without the application of heat? What has happened to the alcohol during the process?

Observations: The reaction would not occur at a perceptible rate without heating. The alcohol evaporates.

b. Shake vigorously in a test tube a mixture of 1 ml of kerosene and 10 ml of distilled water. Describe the result. What are such mixtures called? Now transfer a small portion of the soap you prepared to the test tube and again shake the contents vigorously. Add more soap if necessary. Result? Compare with the initial result. State concisely the action of the soap.

Observations: Vigorous shaking produces a temporary cloudy suspension which quickly separates into two separate layers. Upon shaking with soap a single layer of emulsified kerosene results. The soap acts as an emulsifying agent.

QUESTIONS: 1. Give the name and write the structure representative of the characteristic (functional) groups in the following types of compounds: alcohol, aldehyde, ketone, ether, carboxylic acid.

Alcohol-hydroxyl: —OH

Aldehyde-formyl: —CHO

Ketone-carbonyl: —CO

Ether-oxide: —O—

Carboxylic acid-carboxyl: —COOH

2. Write general formulas for the following: alcohol, aldehyde, ketone, ether, carboxylic acid, ester.

ROH, RCHO, RCOR', ROR', RCOOH, RCOOR'.

FURTHER EXPERIMENTATION: Try the reaction of a small piece of dry sodium (half the size of a small pea), obtained from your instructor, on ethanol and test for hydrogen production.

Try the Beilstein test for the presence of halogens in organic compounds by plunging the heated copper spiral used in Part 2 into a small sample of CCl_4 (representing an alkyl halogenide). **CAUTION:** *Do not inhale the vapors of carbon tetrachloride since they are poisonous.* Reheating the copper spiral produces a characteristic green color in the burner flame. Try testing the CCl_4 for the presence of chloride ion by reacting it with a small quantity of 0.1-*M* $AgNO_3$ (aq) solution.

Try producing Nylon (reference, Morgan, P. W., and Kwolek, S. L., J. Chem. Educ., 36, 182 (1959), *The Nylon Rope Trick.*

Try the polymerization (addition or vinyl type) of styrene by mixing 5 ml of styrene with a small amount of catalyst (paste benzoyl peroxide) and carefully heating the small test tube in a water bath. **CAUTION:** *Dry benzoyl peroxide should not be used because of its explosive nature. Styrene should not be heated with an open flame because of its volatility and combustibility.*

EQUATIONS

1. First step in direct halogenation of methane. $CH_4 + X_2 \rightarrow CH_3X + HX$
2. Oxidation of methanol (hot copper(II) oxide). $CH_3OH + CuO \rightarrow HCHO + Cu + H_2O$
3. Ionization of acetic acid in water. $CH_3COOH + H_2O \rightleftarrows H_3O^+ + CH_3COO^-$
4. Ethyl acetate by esterification. $CH_3COOH + C_2H_5OH \xrightarrow{H_2SO_4} CH_3COOC_2H_5 + H_2O$
5. Aldehyde reaction (Fehling's solution). $RCHO + 2\,CuSO_4 + 5\,NaOH \rightarrow RCOONa + Cu_2O(s) + 2Na_2SO_4 + 3\,H_2O$
6. Saponification of a fat (sodium hydroxide).

$$\begin{matrix} RCOOCH_2 \\ \\ R'COOCH \\ \\ R''COOCH_2 \end{matrix} + 3\,NaOH \rightarrow RCOONa + R'COONa + R''COONa + C_3H_5(OH)_3$$

Experiment 35

NAME . . . . . PERIOD . . . . . DATE . . . . . .

INSTRUCTOR'S COMMENTS . . . . . .

Rate of Chemical Reaction

PURPOSE: To show how the rate of a particular chemical reaction, the decomposition of sodium hypochlorite, may be measured To study the effects of changing the conditions of concentration and temperature on the rate of this reaction.

APPARATUS: Beaker, 600 ml; burner and tubing; clamp, buret; clamp, pinch; cylinders, graduated, 10 ml, 50 ml, 250 ml; flask, Erlenmeyer, 250 ml; flask, Florence, 500 ml; glass bends, 8 cm × 8 cm (2), 8 cm × 20 cm; glass tubing, 5 cm; ring, iron; ring stands (2); rubber connector; rubber tubing, small diameter; stoppers, rubber, 1-hole No. 6, 2-hole No. 6; test tube, small (litmus paper vial will do); thermometer; watch with second hand; wire gauze.

MATERIAL: Ice; graph paper; solutions of cobalt(II) nitrate (0.17 *M*); sodium hypochlorite (5%) (commercial bleach such as Clorox.)

INTRODUCTION: A solution of sodium hypochlorite decomposes in the presence of a catalyst according to the net equation:

$$2OCl^- \rightarrow 2\,Cl^- + O_2 \uparrow$$

Its rate of decomposition is measured in terms of the volumes of oxygen liberated during regular time intervals. Such volumes will be measured by the volumes of water displaced by the liberated oxygen. A catalyst is necessary to speed up the normal, very slow decomposition rate. It is thought to be cobalt(III) oxide, Co_2O_3, which is presumably produced from the standardized amount of cobalt(II) nitrate solution. A negligible quantity of the hypochlorite solution is consumed in the production of the catalyst according to the equation:

$$2Co^{++} + OCl^- + 6H_2O \rightarrow 4H_3O^+ + Cl^- + Co_2O_3$$

The effects of changing the temperature and concentration of the reactant will be determined and the observed results will be tabulated. Graphs will be constructed from the observed, tabulated data.

SUGGESTION: Students should work in pairs. One student times and records results, the other observes the volume of displaced water.

PROCEDURE

1. *General.* The assembled apparatus is shown in Fig. 35-1. Prepare a hot water bath as in Fig. F-6, omitting the watch glass, for Part 3. In general, procedures will parallel Part 2 in dealing with changes in temperatures and concentration. Record all your observations in the proper columns and spaces in the Data Table on page 197.

2. *Rate of reaction at room temperature.* Using the 50-ml graduated cylinder, transfer 25 ml of sodium hypochlorite solution

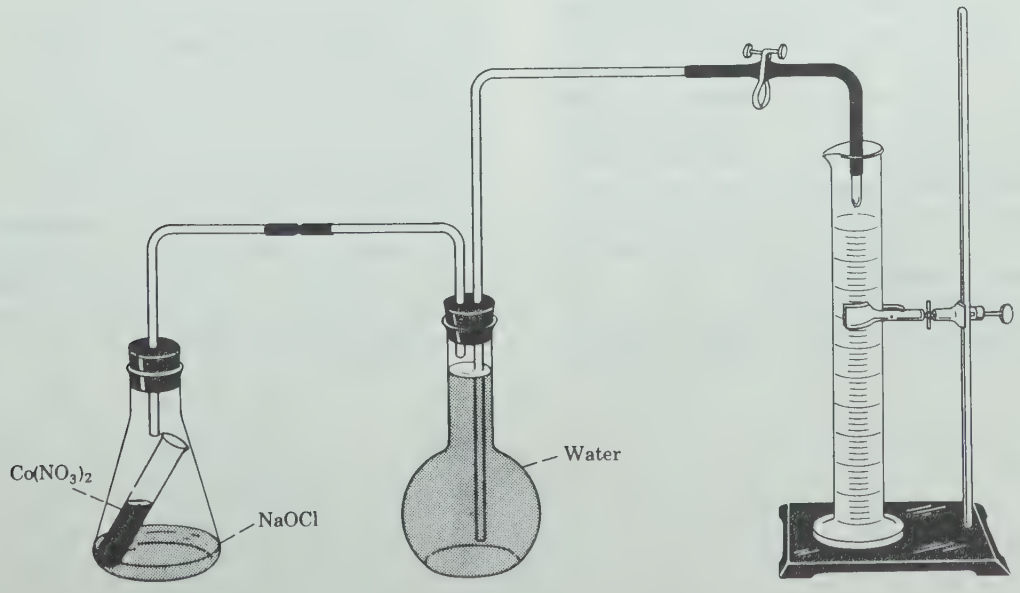


Fig. 35-1

to the Erlenmeyer flask. Take and record the solution temperature. Transfer 5 ml of cobalt(II) nitrate solution to the small test tube. Insert this test tube in the Erlenmeyer flask without spilling. Fill the 500-ml Florence flask with water. Insert the two-hole stopper containing an 8 cm × 8 cm and an 8 cm × 20 cm glass bend in the Florence glask and attach the rubber tubing, pinch clamp, and short glass tube as shown in Fig. 35-1. Open the pinch clamp and blow into the short right-angle bend to fill the delivery tube leading to the graduated cylinder with water. Close the pinch clamp. Insert the second short right-angle bend in the one-hole stopper, and place this stopper in the Erlenmeyer flask. Complete the assembly of the apparatus by connecting the short right-angle bends with a rubber connector. Make sure all stoppers and connections are tight. Remove the pinch clamp. Only a few drops of water will flow into the graduated cylinder if the apparatus is air-tight. Discard this water.

The observer notes the time as the other team member quickly tips over the Erlenmeyer flask so that the cobalt(II) nitrate solution mixes with sodium hypochlorite solution. Hold Erlenmeyer flask at its neck and shake it constantly with a gentle swinging motion throughout the experiment. Observe and record the volume of water displaced every 30 seconds until approximately 200 ml of water (total volume) has been displaced. The timer calls out the time and records the data reported by the observer.

Plot the results on the graph, using times (in minutes) as abscissas and volumes of oxygen in ml as the ordinates.

3. *Rate of reaction at a higher temperature.* Wash out the reaction flask and refill with sodium hypochlorite and test tube containing cobalt(II) nitrate as in Part 2. Place the Erlenmeyer flask with its contents in the hot water bath. Insert a thermometer into the hypochlorite solution. When the temperature of the contents is approximately 10 degrees above room temperature, remove the flask from the bath, reconnect the apparatus, and proceed as in Part 2. Record the results in the Data Table.

4. *Rate of reaction at a temperature below room temperature.* Repeat Part 2 but cool the reaction flask and contents by placing in a beaker of ice water. Take the temperature of the sodium hypochlorite solution, and remove the flask from the ice water bath when the temperature is about 10 degrees below room temperature. Record your results in the Data Table.

5. *Rate of reaction at two-fold dilution.* Wash out the reaction flask and fill as for Part 2. Add 30 ml of distilled water to the sodium hypochlorite solution. Note that when 55 ml of diluted sodium hypochlorite solution is mixed with 5 ml of cobalt(II) nitrate solution, the total volume will be 60 ml instead of the original 30 ml. Repeat Part 2 and record the results.

6. Graph the data obtained for Parts 2-5 and label each of the four curves by the number of the Part which each graph represents.

QUESTIONS: 1. Which numbered curve has the steepest slope? No. 3

2. State the average rate of gas production in ml/min at room temperature.

Student answer.

3. How did the average rate of gas production at room temperature compare with the average rate at (a) approximately 10 C° below room temperature and (b) two-fold dilution? (a) Student answer.

(b) Student answer.

FURTHER EXPERIMENTATION:

(a) Nature of reactants: Test the rate of reaction of Mg, Zn, and Cu in dilute (1:3) hydrochloric acid. (b) Concentration: Use equal masses of Mg in 10 ml of the dilute HCl, and then in 10 ml of the dilute acid produced by dilution to one-half and then to one-quarter of the initial concentrations. The masses of Mg may be wrapped in Cu wire to keep the Mg submerged. Measure the time used for complete consumption of the Mg at the various dilutions.

DATA TABLE

Student answers.

Time interval (minutes)	Volume of Oxygen produced (ml)			
	Room temp. ..... °C	Above room temp. ..... °C	Below room temp. ..... °C	Room temp. and two-fold dilution
0.5	.....	.....	.....	.....
1.0	.....	.....	.....	.....
1.5	.....	.....	.....	.....
2.0	.....	.....	.....	.....
2.5	.....	.....	.....	.....
3.0	.....	.....	.....	.....
3.5	.....	.....	.....	.....
4.0	.....	.....	.....	.....
4.5	.....	.....	.....	.....
5.0	.....	.....	.....	.....
5.5	.....	.....	.....	.....
6.0	.....	.....	.....	.....

NAME ..... PERIOD ..... DATE .....

INSTRUCTOR'S COMMENTS .....

Experiment 36 Course of a Chemical Reactions: Catalysis

PURPOSE: To investigate the decomposition of hydrogen peroxide: (a) the decomposition products; (b) the thermal nature of the reaction; (c) the course of the catalyzed and uncatalyzed reaction.

APPARATUS: Beaker, (2) 400 ml; burner with tubing; clamp, buret; glass tube bend; ring, iron; ringstand; stoppers, rubber, solid No. 2, and a 1-hole No. 2; test tubes, thermometer mounted in stopper; tubing, rubber; wire gauze.

MATERIALS: Hydrogen peroxide solution, manganese dioxide, powder.

INTRODUCTION: Based on the results of the following procedures, you are to attempt to find answers to the problems indicated in the purpose of this experiment and to draw graphs representative of your experimental findings.

PROCEDURE: 1. Add 10 ml of hydrogen peroxide solution to a test tube. Insert the thermometer and record the temperature of the solution. Add about 0.5 gram of manganese dioxide to this solution. Result? Insert the thermometer into the test tube contents and record the temperature after one minute has elapsed. Remove the thermometer and insert a glowing splint into the upper part of the test tube. Result? Explain. Is the decomposition of hydrogen peroxide an endothermic or exothermic reaction. Write a thermochemical equation for the reaction.

Observations: Student answers. .....

.....

.....

.....

a. Initial temperature ..... °C

b. Temperature during the reaction ...

student answer .....

c. What is the thermal nature of this reaction? exothermic .....

d. Name one product of this reaction that you have identified ... Oxygen ...

e. What is the other reaction product? .

water .....

f. Thermochemical equation for the reaction. $2H_2O_2 \rightarrow 2H_2O + O_2(g) + \text{heat}$

2. a. Preheat 200 ml of water in the beaker to approximately 50° C.

b. Fill a test tube with water, stopper it, and place it mouth downward into the second beaker which contains 200 ml of cold water. Remove the stopper under water so that the test tube is ready for gas collection by displacement of water.

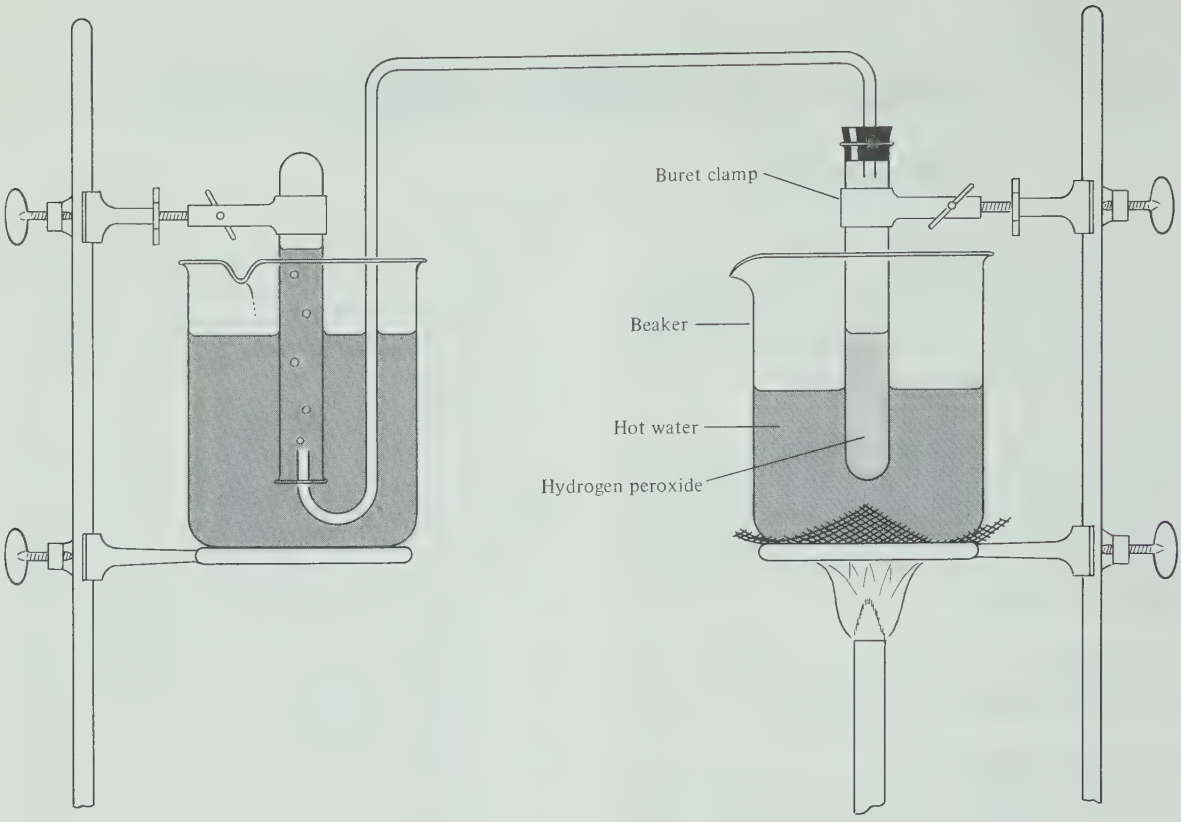
c. Place 20 ml of hydrogen peroxide solution into the generator test tube. Is there any evidence of hydrogen peroxide decomposition at room temperature? Insert the delivery tube assembly into the generator test tube. Place the generator test tube into the hot water and place the end of the delivery tube below the water in the beaker having the collector test tube. Heat the hot water and note the temperature at which gas bubbles start to escape from the hydrogen peroxide. What effect does increasing the temperature have on the rate of chemical reactions? Continue heating the hot water to the temperature range 70–80° C. Do not heat above 80° C. After the first 30 bubbles of gas have escaped, insert the delivery tube into the mouth of the collecting test tube. See Fig. 36-1. Note the time necessary to collect a full test tube of the gas being produced. Remove the test tube, open it, and test the contents with a glowing splint. Result? How does the activation energy of this uncatalyzed reaction compare with that for the catalyzed reaction?

Observations: student answer .....

.....

.....

.....



QUESTIONS:

1. Is there any evidence of H_2O_2 decomposition at room temperature? No

2. What is the temperature at which decomposition starts? Student answer

3. What is the effect of increasing temperature on the rate of a chemical reaction? Increasing temperature increases the rate of a chemical reaction.

4. Why is it necessary to permit some gas to escape from the delivery tube before starting the gas collection? To obtain a relatively pure sample of O_2 since air is also being displaced.

5. How does the activation energy required for the catalyzed reaction compare with that for the uncatalyzed reaction? The activation energy for the catalyzed reaction is less than that for the uncatalyzed reaction.

6. On graph paper, construct curves representing the course of both the catalyzed and uncatalyzed reactions. Use dotted lines for the catalyzed reaction. Use arrows with labels to show the relative activation energies.

FURTHER EXPERIMENTATION: The addition of Fe^{+++} ion catalyzes H_2O_2 decomposition. The addition of phosphate ion, which combines with the Fe^{+++} ion, prevents the catalytic action. Try adding Fe^{+++} to H_2O_2 solution. Then add phosphate ion. Reverse the procedure.

Experiment 37

NAME ..... PERIOD ..... DATE .....

INSTRUCTOR'S COMMENTS .....

Equilibrium

PURPOSE: To examine the effect of concentration on various equilibria.

APPARATUS: Beaker, 250 ml; burner and tubing; clamp, test tube; funnel; medicine dropper; stirring rod, glass; ring, iron; ring stand; rubber stopper, solid No. 2; test tubes.

MATERIAL: Ammonium chloride; ammonium thiocyanate; iron(III) chloride; iron (III) nitrate; silver acetate; silver nitrate; sodium acetate; sodium chloride; sodium nitrate; filter paper; pH paper (Hydrion A and B); acetic acid, dil. (1.0*M*); hydrochloric acid (12*M*); solutions of barium chloride (0.5*M*); calcium chloride(0.5*M*); iron(III) chloride (1*M*); potassium thiocyanate (1*M*); silver acetate (saturated); sodium sulfate (1*M*).

INTRODUCTION: Chemical reactions in which a product is essentially un-ionized, is given off as a gas, or is precipitated, may be thought of as running to completion and are commonly referred to as *end reactions*. Many of the reactions you have studied are of importance because they run to completion, yielding products which are easily recovered. Many other reactions, however, do not run to completion. The products, if formed at all, do not leave the fields of action but remain in contact and react to re-form the original reactants. Under suitable conditions, both reactions may proceed at the same speed. An equilibrium is thus established. Both the forward and the reverse actions continue with no net change in the quantities of either reactants or products.

An equilibrium exists in a saturated solution between undissolved particles and particles in solution. An ionic equilibrium may be established between un-ionized molecules of a molecular solute and the ions of the solution, or between undissociated ions of an ionic solute and the ions in solution. An equilibrium is affected by both concentration and temperature. By the principle of Le Chatelier, we know that a system in equilibrium tends to shift so as to relieve the stress placed upon it by a change in concentration or temperature.

PROCEDURE: 1. Solution equilibrium. Add Rapidly 10 ml of calcium chloride solution to 5 ml of sodium sulfate solution in a Pyrex test tube. Shake. Result? Write the equation for this reaction in ionic form. Use the Table of Solubility of Salts in the Appendix, if necessary, to identify the precipitate. Now add dropwise 5 ml more of the calcium chloride solution, observing closely to see whether the precipitate continues to form. From the quantities of the two solutions used and their molarities, which ions have been added to the mixture in excess? Heat the tube just to boiling and set it aside to allow the precipitate to settle. This should require approximately 30 minutes; in the meantime, proceed with Parts 2 and 3.

After the precipitate has settled, add a drop of calcium chloride solution to the clear supernatant liquid, using a clean, dry medicine dropper. If no precipitate forms where the drop contacts the liquid, you may be sure that calcium chloride is present in excess in the solution. Decant this clear liquid into a second test tube. (if the mixture is still turbid, it may be filtered.) To the filtrate (or decanted liquid), add 3 or 4 ml of barium chloride solution. Result? Refer to the equation you balanced after the initial precipitation. Which ion, when paired with the barium ion just added, will yield a precipitate? The initial precipitate must have separated from a solution that was saturated with respect to *which* pair of ions? Write the equation for the equilibrium between this saturated solution and the *initial* precipitate.

Observations: A white precipitate forms.

$Ca^{++}(aq) + SO_4^{--}(aq) \rightarrow CaSO_4(s)$

Initially, the sulfate ion was in excess. Upon addition of 5 additional ml of calcium ion, neither ion should be present in excess. A precipitate forms.

The sulfate ion, if present, will yeild a precipitate with the barium ion.

The separated solution was saturated with respect to calcium and sulfate ions.

$CaSO_4(s) \rightleftarrows Ca^{++}(aq) + SO_4^{--}(aq)$

2. *Common-ion affect.* Add a very small amount of solid silver acetate to 15 ml of saturated silver acetate solution. Stopper the test tube and shake vigorously for about one minute. If the solid portion dissolves, add more and repeat until satisfied that your solution is saturated, which will be indicated when the solid silver acetate fails to dissolve. Write the equation for the solution equilibrium now assured. Filter and divide equally among 3 dry test tubes. Add to these three samples, respectively, 0.5 g solid silver nitrate, 0.5 g solid sodium acetate, and 0.5 g solid sodium nitrate. Shake each. Result? To which tubes have ions been added that are common to an ion already present? Refer to the equilibrium equation you have just written and explain the result observed in each tube.

Observations: $AgC_2H_3O_2(s) \rightleftarrows Ag^+(aq) + C_2H_3O_2^-(aq)$. Solid silver nitrate and solid sodium acetate produce precipitates, since they are added to a solution which contains a common ion. Increasing the concentration of either the silver or acetate ion causes the K_{sp} of silver acetate to be exceeded with consequent precipitation. Sodium nitrate produces no precipitation because no common ion is added.

3. *Le Chatelier's principle:* Set up 4 test tubes each containing 5 ml of 1.0-*M* acetic acid. Keep test tube No. 1 as the control. To test tube No. 2 add 1 g of solid sodium acetate. Stir the contents thoroughly. To test tube No. 3 add 1 ml of 12 *M* HCl and mix thoroughly. To test tube No. 4 add 1 g of solid NaCl and mix thoroughly. Place strips of pH paper on a clean dry glass plate. Using a clean stirring rod, first test the contests of test tube No. 1 by dipping the stirring rod in the solution and touching to the pH paper until you have determined the pH of the 1.0-*M* acetic acid. Repeat this operation for the contents of each of the other three test tubes. Report the pH in the Data Table. Complete the rest of the Data Table by indicating how the results affect the molar concentrations of each of the species present in the original solution of acetic acid. Use the terms: *increases*, *decreases*, *remains the same*. In which test tubes was a stress applied to the equilbirium system? How would you identify this type of stress?

Observations: A stress was applied to test tubes 2 and 3. The stress was one of increased concentration of acetate ion in test tube 2 and hydronium ion in test tube 3.

4. *Complex-ion equilibrium.* Mix approximately 1 ml each of the solutions of iron(III) chloride and potassium thiocyanate in a 250-ml beaker. Result? The Fe^{+++} ion and the CNS^- ion form the complex $FeCNS^{++}$ ion which shows a deep-red color. Assuming the $FeCNS^{++}$ ion to be only slightly ionized at this concentration to form Fe^{+++} ions and CNS^- ions, write the equation for the ionic equilibrium. Now dilute with water (100 ml or more) to give a color of light red to orange-yellow. Half fill 4 test tubes and add, respectively, 0.5 g of the following solid reagents: iron(III) nitrate, iron(III) chloride, ammonium thiocyanate, and ammonium chloride. Results? Explain your observations in terms of shifts in the equilibrium using the equation you have written.

Observations: The deep-red color of the complex $FeCNS^{++}$ ion results. $Fe^{+++} + CNS^- \rightleftarrows FeCNS^{++}$. Addition of the iron(III) and of the thiocyanate solutions causes the color of the diluted solution to deepen due to the equilibrium shift toward the formation of more of the complex $FeCNS^{++}$.

DATA TABLE Student answers.

	pH	$HC_2H_3O_2$	H_3O^+	$C_2H_3O_2^+$
#1 1.0-*M* $HC_2H_3O_2$	.....	xxxxxx	xxxxxx	xxxxxx
#2 plus solid $NaC_2H_3O_2$	.....	.....	.....	.....
#3 plus 12-*M* HCl	.....	.....	.....	.....
#4 plus solid NaCl	.....	.....	.....	.....

FURTHER EXPERIMENTATION: Prepare a saturated solution of sodium chloride and filter it. To 5 ml portions of the filtrate add separately: 1 ml of concentrated HCl; 1 ml of saturated NaOH solution; two pellets of solid sodium hydroxide; 1 ml of saturated solutions of sodium nitrate, calcium chloride, and potassium nitrate. Look for evidences of the production of a precipitate in each case, and explain the results in terms of the effect of ions on a solution equilibrium of a salt.

Use Welch #4226 Temperature Equilibrium Tubes for the $2NO_2 \rightleftarrows N_2O_4$ system by immersing in (a) hot water, (b) ice-water mix, (c) either salt-ice or Dry Ice-acetone. As a project students may prepare their own tubes, Reference: *Chemistry*, 45, Jan., (1972), "Making Tubes for NO_2-N_2O_4 Equilibrium Reaction."

Prepare a saturated solution of CO_2 or use soda water to test effect of temperature rise on equilibrium system by testing with litmus or pH paper. Heat gently, increase temperature to boil out, making indicator tests. Refer to heat of solution of CO_2, Modern Chemistry, 1974, Chapter 12.

QUESTIONS: 1. How is a saturated solution defined in terms of an equilibrium condition? A saturated solution is one in which the dissolved and undissolved solutes are in equilibrium.

2. How does the addition of sodium acetate to a dilute solution of acetic acid affect the pH of the solution? Explain. The pH increases due to the common ion effect. The ionization of acetic acid is reduced and the concentration of the hydrogen ion is decreased.

3. How would the addition of ammonium chloride to a dilute ammonia-water solution affect the hydroxide ion concentration of the solution? Explain. The hydrogen ion concentration is reduced. The addition of NH_4^+ ion reduces the tendency for the ammonia water to ionize.

4. How would the addition of sodium hydroxide to a dilute solution of ammonia-water affect the concentrations of (a) the hydroxide ion, (b) the ammonium ion initially present? Explain. (a) Increase the concentration of the OH^- because NaOH is a strong base; (b) decrease the concentration of the ammonium ion because the tendency for the ammonia-water to ionize decreases.

Experiment 38

NAME ..... PERIOD ..... DATE .....

INSTRUCTOR'S COMMENTS .....

Solubility-Product Constants

PURPOSE: To show how the difference in the magnitudes of solubility-product constants of characteristic precipitates may be used to separate effectively a mixture of related ions.

APPARATUS: Funnel; graduated cylinder; ring, iron; ring stand; test tubes; wash bottle.

MATERIAL: Ethanol (denatured alcohol will do); filter paper; litmus paper; acetic acid, dilute (1:4); ammonia-water solution, dilute (1:4); hydrochloric acid, concentrated; solutions of barium chloride (0.25 *M*); potassium chromate (0.25 *M*); strontium chloride (0.25 *M*).

INTRODUCTION: The chromates of barium and strontium are insoluble according to the following data:

Salt	Ion Product	K_{sp}
$BaCRO_4$	$[Ba^{++}]\ [CrO_4^{--}]$	2×10^{-10}
$SrCrO_4$	$[Sr^{++}]\ [CrO_4^{--}]$	3.6×10^{-5}

By controlling the concentration of chromate ion added to a solution containing barium and strontium ions in identical concentration, it is possible to cause almost complete precipitation of the less soluble barium chromate. Under the conditions, strontium chromate is not precipitated and strontium ions remain in solution.

In a solution of chromate ions, such as that of potassium chromate, the existent equilibrium is

$$Cr_2O_7^{--} + 3H_2O \rightleftarrows 2CrO_4^{--} + 2\,H_3O^+$$

For this system

$$K = \frac{[CrO_4^{--}]^2\ [H_3O^+]^2}{[Cr_2O_7^{--}]} = 2.4 \times 10^{-15}$$

According to the law of mass action, increasing the hydronium ion concentration results in a decrease in the chromate ion concentration. In some qualitative analysis procedures, the chromate ion concentration is maintained at about 5×10^{-4} *M* by adjusting the pH to 5. This is achieved by using a solution of chromate ion containing acetic acid and ammonium acetate. This concentration of chromate ions is sufficient to reduce the concentration of barium ion from 0.1 *M* to 0.0001 *M* but is insufficient to exceed the K_{sp} for strontium chromate from a 0.1-*M* solution of this ion. In this simplified procedure, acetic acid is used in sufficient concentration to achieve a functional separation of barium and strontium ions. Ethanol is added to a solution of strontium chromate from which barium chromate has been precipitated. This lowers the K_{sp} of strontium chromate so that its ion-product is exceeded and precipitation occurs.

SUGGESTION: The instructor may wish to have the students calculate (a) the concentration of chromate ion required to reduce the concentration of barium ion to 1×10^{-4} *M* and (b) the value of the ion-product for strontium chromate at this concentration of chromate ion for a solution which contains 0.1-*M* strontium ion. Flame tests for barium and strontium may also be used at appropriate places in this experiment according to directions given in Part 2, Experiment 45, Flame Tests.

PROCEDURE: 1. Add 5 ml of acetic acid to 5 ml of barium chloride solution. Stir. Add 5 ml of potassium chromate solution. Stir. Result? Explain. Filter, and wash the precipitate twice with small additions of water from a wash bottle. Puncture the filter cone and wash one-half of the precipitate into a test tube with 5 ml of water. Use another 5 ml of water to wash the remaining precipitate into a second test tube. Add 1 ml of concentrated hydrochloric acid to the mixture in one test tube. Add 5 ml of acetic acid to the mixture in the other test tube. Stir each. Results? Explain.

Observations: A yellow precipitate of $BaCrO_4$ results because the K_{sp} has been exceeded. This precipitate does not dissolve in acetic acid but does dissolve in hydrochloric acid being converted to the soluble $BaCr_2O_7$ due to the high concentration of hydronium ion.

2. Add 5 ml of acetic acid to 5 ml of strontium chloride solution. Stir. Add 5 ml of potassium chromate solution. Stir. Result? Explain. Add ammonia-water with stirring

until the solution gives an alkaline reaction with litmus paper. Result? Explain. Mix 5 ml of this solution with 5 ml of ethanol. Result? Filter and wash one-half of the precipitate into each of two test tubes as in Part 1. Add 1 ml of concentrated hydrochloric acid to the precipitate in one test tube and 5 ml of acetic acid to the precipitate in the second test tube. Stir each. Results? Explain.

Observations: A precipitate does not result since the K_{sp} of $SrCrO_4$ is not exceeded. Addition of ammonia-water results in changing the orange-red dichromate to the yellow $SrCrO_4$ since the concentration of chromate ion is increased by making the solution alkaline. Addition of alcohol results in the complete precipitation of $SrCrO_4$. The precipitate dissolves in HCl but not in acetic acid because the chromate ion is changed to dichromate ion in the strong acid, HCl.

3. Mix 4 ml of strontium chloride with 2 ml of barium chloride. Add 6 ml of acetic acid. Stir. Add 6 ml of potassium chromate solution. Stir. Using Hydrion paper and drops of ammonia-water solution, adjust the pH to between 4 and 5. Shake vigorously. Result? Filter and save the filtrate for Part 4. As in Part 1, wash the precipitate, puncture the filter cone and wash one-half of the precipitate into each of two test tubes. Add 1 ml of concentrated hydrochloric acid to one portion of precipitate and 5 ml of acetic acid to the other portion. Stir each. Results? Explain. Identify the precipitate.

Observations: Yellow $BaCrO_4$ is precipitated. It dissolves in HCl. The presence of barium ion may be confirmed by a flame test.

4. Add ammonia-water solution dropwise to 5 ml of the filtrate from Part 3 until the mixture just turns red litmus to a definite blue. Result? Explain. Mix 5 ml of this solution with 5 ml of ethanol. Result? Filter. As in Part 3, wash one-half of the precipitate into each of two test tubes. Add 1 ml of concentrated hydrochloric acid to one portion and 5 ml of acetic acid to the other portion. Stir each. Results? Identify the precipitate.

Observations: The precipitation of $SrCrO_4$ begins (and is completed by subsequent alcohol addition) since the concentration of chromate ion is sufficient to exceed the K_{sp} of $SrCrO_4$. The yellow precipitate dissolves in HCl and a flame test may be used to confirm the strontium.

FURTHER EXPERIMENTATION: Try precipitating ions of both metals from a mixed solution, made alkaline with ammonia-water using a solution of ammonium carbonate, and dissolve the precipitate in acetic acid before going on to Parts 3 and 4. This procedure is used in qualitative analysis to separate barium, strontium, and calcium from other ions. Try precipitating barium sulfate from a solution of barium ion, acidified with 1 ml of hydrochloric acid. This procedure is often used to identify barium in conjunction with a flame test for barium ion.

QUESTIONS: 1. Explain why barium chromate dissolves in hydrochloric acid. The high concentration of hydronium ion reduces the concentration of CrO_4^{--} ion by converting it to $Cr_2O_7^{--}$ (See equation 1 in Introduction). The decrease in the concentration of the CrO_4^{--} ion causes the $BaCrO_4$ to dissolve.

2. Refer to the equation in the Introduction and explain the effect of addition of hydroxide ion (addition of ammonia-water solution) on the concentration of chromate ion. Addition of hydroxide ion increases the concentration of the CrO_4^{--} ion by decreasing the concentration of the hydronium ion.

NAME ..... PERIOD ..... DATE .....
INSTRUCTOR'S COMMENTS .....

Experiment 39 Hydrolysis

PURPOSE: To test the water solutions of some salts to discover which are not neutral, and to explain why this phenomenon occurs.

APPARATUS: Dish, evaporating; glass plates; test tubes.

MATERIAL: Aluminum sulfide; Hydrion papers, full range; litmus papers, red and blue; solutions of: aluminum sulfate (0.02 *M*); ammonium chloride (0.1 *M*); copper(II) sulfate (0.05 *M*); potassium carbonate (0.05 *M*); potassium chloride (0.1 *M*); potassium nitrate (0.1 *M*); sodium acetate (0.1 *M*); sodium hydrogen carbonate (0.1 *M*); sodium phosphate (0.03 *M*); sodium sulfate (0.05 *M*); zinc chloride (0.05 *M*).

INTRODUCTION: Hydrolysis is a process which involves the reaction of water with the ions of certain salts. In the slight ionization of water, equilibrium is quickly achieved and the concentrations of hydronium ions and hydroxide ions are very low.

$$2H_2O \rightleftarrows H_3O^+ + OH$$

Any reaction which removes either of the ions will speed the reaction producing these ions. Thus hydrolysis always shifts the equilibrium to the right, resulting in an increase in the concentration of the ion not removed. If hydrolysis removes hydronium ions by the formation of a weak acid, the solution will be basic due to the increase in hydroxide-ion concentration. If hydrolysis removes OH; ions by the formation of a weak hydroxide, the solution will be acidic due to the increase in the H_3O^+-ion concentration. If a weak acid and an equally weak hydroxide are formed by hydrolysis, the solution will remain neutral and the hydrolysis may be complete.

PROCEDURE: 1. Place 1-ml portions of each of the following solutions in separate labeled test tubes: (a) sodium chloride, (b) sodium sulfate, (c) potassium chloride, (d) potassium nitrate. Test each with strips of red and blue litmus, and with Hydrion papers. If short range indicator papers are available, close approximations of the pH of each solution may be possible. Record your results in the Data Table including the approximate pH.

DATA TABLE

Solution tested	Indicator color		Approximate pH	Approximate	
	Litmus	Hydrion		$[H_3O^+]$	$[OH^-]$
NaCl	No change.		7	10^{-7}	10^{-7}
Na_2SO_4	No change.		7	10^{-7}	10^{-7}
KCl	No change.		7	10^{-7}	10^{-7}
KNO_3	No change.		7	10^{-7}	10^{-7}
$NaC_2H_3O_2$	Red to blue.	Student answers.	Student answers.	Student answers.	Student answers.
$NaHCO_3$	Red to blue.				
Na_3PO_4	Red to blue.				
K_2CO_3	Red to blue.				
NH_4Cl	Blue to pink.				
$Al_2(SO_4)_3$	Blue to red.				
$CuSO_4$	Blue to red.				
$ZnCl_2$	Blue to red.				

2. Test 1-ml portions of each of the following solutions as in Part 1: (a) sodium acetate, (b) sodium hydrogen carbonate, (c) sodium phosphate, (d) potassium carbonate. Record your results in the Data Table.

3. Test 1-ml portions of each of the following solutions as in Part 1: (a) ammonium chloride, (b) aluminum sulfate, (c) copper (II) sulfate, (d) zinc chloride. Record your results in the Data Table. Now complete the last two columns of the Data Table.

4. Put a small lump of aluminum sulfide in an evaporating dish in the hood or other well ventilated place. Add water to the dish. Identify the gas that forms by its odor, and examine the precipitate. Why may this hydrolysis action be considered to be complete?

Observations: The gas has the rotten-egg odor of H_2S. The gelatinous precipitate is $Al(OH)_3$. The hydrolysis runs to completion because both products are removed from solution.

QUESTIONS: 1. Why do the solutions of some of the salts tested have a basic reaction? Equilibrium is distributed in the direction of OH^- ion formation due to the formation of slightly ionized acid molecules.

2. Why do the solutions of some of the salts tested have an acidic reaction? Equilibrium is disturbed in the direction of H_3O^+ ion formation due to the formation of either slightly ionized base molecules or of hydrated metallic ions (which may denote protons to water molecules thus forming H_3O^+ ions).

3. Write the equation for the net reaction in the hydrolysis of the diluate sodium hydrogen carbonate solution.

$HCO_3^- + H_2O \rightarrow H_2CO_3 + OH^-$

4. If the reaction to the right (in your equation for Question 3) were complete, what would be the OH^- ion concentration?

If the forward reaction were complete, the OH^- concentration would be 10^{-1} mole (g-ion) per liter.

5. What was the experimental OH^- ion concentration of the sodium hydrogen carbonate solution as indicated by the Hydrion paper? Student answer.

6. What percent of the sodium hydrogen carbonate is hydrolyzed as shown by your experimental results?

$$\% \text{ hydrolyzed} = \frac{\text{Experimental } [OH^-]}{[OH^-] \text{ if hydrolysis complete}} \times 100\%$$

$$\% \text{ hydrolyzed} = \frac{\qquad}{\qquad} \times 100\%$$

$$= \qquad \%$$ Student answer.

7. Considering both positive and negative ions, which of the two salt solutions, $NaC_2H_3O_2$ or $NH_4C_2H_3O_2$, would you expect to be hydrolyzed more completely? Why? The ammonium acetate would hydrolyze more completely because both products would be weakly ionized.

EQUATIONS: Complete these equations for cation or anion hydrolysis.

1. $NH_4^+ + H_2O \rightleftarrows H_3O^+ + NH_3$
2. $Al(H_2O)_6^{+++} + H_2O \rightleftarrows Al(H_2O)_5OH^{++} + H_3O^+$
3. $Cu(H_2O)_4^{++} + H_2O \rightleftarrows Cu(H_2O)_3OH^+ + H_3O^+$
4. $CO_3^{--} + H_2O \rightleftarrows HCO_3^- + OH^-$
5. $C_2H_3O_2^- + H_2O \rightleftarrows HC_2H_3O_2 + OH^-$

NAME . . . . . PERIOD . . . . . DATE . . . . . .
INSTRUCTOR'S COMMENTS . . . . . .

Experiment 40 Equilibrium and Complex Ions

PURPOSE: To show how variation in pressure, temperature, and concentration affect various equilibrium systems, mainly through the study of reactions involving consideration of the equilibrium constants of complex ions and the solubility product constant of related precipitates.

APPARATUS: Balance, platform; glass rod; graduated cylinder; medicine droppers; test tubes, small, Pyrex.

ADDITIONAL APPARATUS for INSTRUCTOR'S EXPERIMENT: Beaker, (400 ml); burner and tubing; clamp, buret; flask, suction; rubber stopper, No. 7; tubing, rubber, suction; vacuum pump (or aspirator).

MATERIAL: Ammonia-water solution, dilute; hydrochloric acid, concentrated, (12 *M*); sodium fluoride; solutions of: cobalt(II) chloride (0.1-*M*); cobalt(II) chloride, saturated; copper(II) sulfate (0.1 *M*); iron(III) chloride (0.1 *M*); potassium thiocyanate (0.1 *M* in acetone.).

ADDITIONAL MATERIALS for INSTRUCTOR'S EXPERIMENT: Ethanol; ice; hydrochloric acid, dilute, (0.1 *M*); phenolphthalein; sodium chloride; sodium hydrogen carbonate.

INTRODUCTION: Transition metals such as iron, copper, and cobalt form complex ions which, unlike familiar polyatomic ions such as the NO_3^- ion, $SO_4^=$, ion and NH_4^+ ion, ionize slightly into simpler fragments (ions). This ionization is slight and its extent is indicated by the magnitudes of the ionization constants. For the tetraamminecopper(II) ion, $Cu(NH_3)_4^{++}$, the ionization constant is 5×10^{-14}.

$$Cu(NH_3)_4^{++} \rightarrow Cu^{++} + 4NH_3$$

$$K_i = \frac{[Cu^{++}][NH_3]^4}{[Cu(NH_3)_4^{++}]} = 5 \times 10^{-14}$$

The first addition of ammonia-water solution to a solution of the copper(II) ion results in the precipitation of the pale blue copper(II) hydroxide. The concentration of hydroxide ion is large enough for the K_{sp} of the hydroxide to be exceeded.

$$Cu(H_2O)_4^{++} + 2OH^- \rightarrow Cu(H_2O)_2(OH)_2\ (s) + 2H_2O$$

$$K_{sp} \text{ of } Cu(H_2O)_2(OH)_2 = [Cu(H_2O)_4^{++}][OH^-]^2 = 5.6 \times 10^{-20}$$

Further addition of ammonia-water solution causes the precipitate to dissolve with the formation of the dark blue tetraaminecopper(II) complex.

$$Cu(H_2O)_2(OH)_2 + 4NH_3 \rightarrow Cu(NH_3)_4^{++} + 2H_2O + 2OH^-$$

This shows the effect of the availability of a sufficiently large supply (concentration) of the coordinating group. For the overall reaction,

$$Cu(H_2O)_4^{++} + 4HN \rightleftarrows Cu(NH_3)_4^{++} + 4H_2O$$

the equilibrium constant is large, approximately 10^{12}.

$$K_{eq} = \frac{[Cu(NH_3)_4^{++}]}{[Cu(H_2O)_4^{++}][NH_3]^4} = 10^{12}$$

In one part of this experiment you will perform this sequence of operations. You will then add concentrated hydrochloric acid dropwise to this system to study the effects of increasing concentration of hydronium and of chloride ions. This leads to the stepwise production of 1. the regenerated copper(II) hydroxide, 2. the hydrated blue copper(II) ion, and 3. the green-yellow tetrachlorocuprate(II) ion, $CuCl_4^{--}$.

1. $Cu(NH_3)_4^{++} + 2H_3O^+ + 2H_2O \rightarrow Cu(H_2O)_2(OH)_2\ (s) + 4NH_4^+$
2. $Cu(H_2O)_2(OH)_2 + 2H_3O^+ \rightarrow Cu(H_2O)_4^{++} + 2H_2O$
3. $Cu(H_2O)_4^{++} + 4Cl^- \rightarrow CuCl_4^{--} + 4H_2O$

SUGGESTION: Set up the Instructor Experiment showing the effect of pressure on equilibrium at the beginning of the period and have students view the results during the period. The effect of temperature on equilibrium may be investigated at a strategic time, probably toward the end after students have experimented with Co(II) complexes.

PROCEDURE: 1. *Complex ions of Cu(II).* Add dilute ammonia-water dropwise to 5 ml of the solution of copper(II) sulfate until the pale blue precipitate of copper(II) hydroxide results. Record the number of drops required in the Data Table. Continue the addition of the ammonia-water solution stirring continuously until the deep blue solution of tetraamine copper(II) results. Record the additional number of drops re-

DATA TABLE

Reagent	Number of drops added	Formula of product
$NH_3(aq)$	Student answers.	
$NH_3(aq)$		
HCl		
HCl		
HCl		

quired. Add hydrochloric acid drop by drop with stirring until the copper(II) hydroxide is regenerated. Record the number of drops required. Continue the addition of hydrochloric acid until the blue color of the hydrated Cu(II) ion is produced. Record the number of drops required. Continue the addition of hydrochloric acid until the green-yellow tetrachlorocuprate(II) ion is produced. Record the number of drops used.

2. *Complex ions of Co(II).* Add hydrochloric acid dropwise to 5 ml of the saturated cobalt chloride solution until a perceptible color change is produced. Result? How many drops are required to change the $Co(H_2O)_6^{++}$ ion to the $CoCl_4^{=}$ ion? Add the dilute solution of cobalt chloride dropwise to 5 ml of the potassium thiocyanate solution (acetone) until the first perceptible color change occurs. Result? Continue the addition until a deeper color occurs. Result? This color is characteristic of the hexathiocyanatocobaltate(II) complex, $Co(SCN)_6^{-4}$. Explain the deepening of the color in terms of concentration of the hexaaquocobalt(II) ion, $Co(H_2O)_6^{++}$. Continue the addition of the cobalt chloride solution until another definite color change occurs. Result? Explain in terms of the changing concentration of water in the system.

Observations: The pink $Co(H_2O)_6^{++}$ changes to the blue $CoCl_4^{--}$ ion: $[Co(H_2O)_6]^{++} + 4Cl^- \rightarrow [CoCl_4]^{--} + 6H_2O$

The thiocyanate solution first turns blue as the increasing concentration of the Co^{++} ion results in the formation of the blue $[Co(SCN)_6]^{-4}$ ion. The color may become deeper blue and then changing back to pink $[Co(H_2O)_6]^{++}$ complex as the concentration of water due to successive additions increases:

$Co(H_2O)_6^{++} + 6SCN^- \rightleftarrows [Co(SCN)_6]^{-4} + 6H_2O$

3. *Identifying the Co(II) ion in the presence of Fe(III) ion.* Add 1 ml of iron(III) chloride solution to 5 ml of the acetone solution of potassium thiocyanate. Result? This identifies the hexathiocyanatoferrate(III) ion, $Fe(SCN)_6^{-3}$. Mix 2 ml each of the solutions of iron(III) chloride and cobalt(II) chloride. Add 2 ml of this mixture to 5 ml of the acetone solution of potassium thiocyanate. Result? Add 0.5 g of solid sodium fluoride to the remaining 2 ml of the mixture of Co(II) and Fe(III) ions. Stir. Result? This result is due to the formation of the hexafluoroferrate(III) ion, FeF_6^{-3}. Then add this solution dropwise to 5 ml of the acetone solution of potassium thiocyanate until the color change establishing the presence of the Co(II) ion in the mixture occurs. Result? The K_i for the hexathiocyanatoferrate equilibrium is 8×10^{-3}. How does the K_i for the hexafluoroferrate equilibrium compare in magnitude with the K_i for the hexathiocyanatoferrate equilibrium?

Observations: Note to teacher: The blood-red solution resulting is due to the complex ion $[Fe(SCN)_6]^{-3}$ which in simpler form may be written as $[FeSCN]^{++}$ (See Experiment 52).

The mixture produces the blood-red color identifying the Fe^{+3} ion and preventing the development of the blue complex of Co^{++}.

The addition of the NaF to the mixture results in the formation of the very stable $[FeF_6]^{-3}$ ion and the red color is destroyed. Upon addition of the resultant solution to the acetone solution of KSCN, the blue color of the CO^{++} complex develops. The K_i for the hexafluoroferrate(III) ion is smaller than that for the hexacyntoferrate ion.

4. INSTRUCTOR EXPERIMENT. *a.* Effect of pressure. Put 250 ml of saturated sodium hydrogen carbonate solution in a suction flask. Add several drops of phenolphthalein solution. If a pink color develops add hydrochloric acid (dilute) dropwise until the color just disappears. Stopper the suction flask and attach the side arm to a vacuum pump or aspirator. Observe periodically until the solution turns pink. Have the students explain on the basis of the following equilibrium.

$$HCO_3^- \rightleftarrows OH + CO_2$$

b. Effect of temperature. Half-fill a large Pyrex test tube with the saturated solution of cobalt(II) chloride. Heat this until the contents of the test tube undergo a color change. Have students explain on the basis of the equilibrium

$$Co(H_2O)_6^{++} + 4Cl^- \rightleftarrows CoCl_4^{--} + 6H_2O$$

Place the hot test tube in an ice-sodium chloride mixture so that the bottom half of the solution cools in the ice-salt mixture. Have students note the results and explain.

c. You may wish to dissolve $CoCl_2 \cdot 6H_2O$ in ethanol to show how a solvent, other than water, may affect an equilibrium. You may also wish to demonstrate the use of cobalt(II) chloride in "invisible" ink.

Experiment 41

NAME ..... PERIOD ..... DATE .....

INSTRUCTOR'S COMMENTS .....

Oxidation-Reduction Reactions

PURPOSE: To study some typical oxidation-reduction reactions, and to determine the relative strengths of some oxidizing and reducing agents.

APPARATUS: Balance, platform; forceps; medicine dropper; test tubes; wire hook.

MATERIAL: Strips 1 × 5 cm of copper, lead, silver, zinc; iron filings; iron(II) sulfate; potassium permanganate; sand paper; hydrochloric acid, concentrated; sulfuric acid, concentrated; solutions of: copper(II) nitrate (0.1 *M*), iron(III) chloride (0.1 *M*), lead(II) nitrate (0.1 *M*), silver nitrate (0.1 *M*), tin(II) chloride (0.1 *M*), zinc nitrate (0.1 *M*).

INTRODUCTION: Substances which lose electrons during chemical action are said to be oxidized. Those which gain electrons are said to be reduced. If one reactant gains electrons, another must lose an equal number. Thus oxidation and reduction actions must occur simultaneously and to a comparable degree.

The stronger the tendency of an oxidizing agent to gain electrons, the greater is its strength. The weaker the tendency for a reducing agent to hold electrons, the greater is its strength as a reducing agent. Thus the silver ion, Ag^+, has a strong tendency to acquire an electron to form the silver atom, Ag. The Ag^+ ion is a weak oxidizing agent.

PROCEDURE: 1. Add 5 ml of silver nitrate solution to a test tube and insert a strip of brightened copper. To 5 ml of copper(II) nitrate solution in a second test tube, add a strip of silver foil. After a few minutes, examine both pieces of metal. Results? Where a reaction has occurred, write the balanced electronic equations for the oxidation and the reduction. Then write the ionic equation for the net reaction. Which metal is the stronger reducing agent? Which metallic ion is the stronger oxidizing agent?

Observations: Crystals of Ag form on the Cu strip. No evidence of reaction between Ag and $Cu(NO_3)_2$ solution. $Cu - 2e^- \rightarrow Cu^{++}$; $Ag^+ + e^- \rightarrow Ag$; $Cu + 2Ag^+ \rightarrow Cu^{++} + 2Ag$. Cu is the stronger reducing agent, Ag^+ is the stronger oxidizing agent.

2. Place a strip of brightened zinc in 5 ml of copper(II) nitrate solution. Insert a strip of copper in 5 ml of zinc nitrate solution. Write the balanced electronic equations for both oxidation and reduction actions where a reaction occurs. Write the ionic equation for the net reaction. Which metal is the stronger reducing agent? Which metallic ion is the stronger oxidizing agent? What are the relative strengths of the three metals tested so far as reducing agents. What are the relative strengths of the three metallic ions as oxidizing agents?

Observations: Cu deposits on the Zn strip. $Zn - 2e^- \rightarrow Zn$; $Cu^{++} + 2e^- \rightarrow Cu$; $Zn + Cu^{++} \rightarrow Zn^{++} + Cu$. Zn is the stronger reducing agent, Cu^{++} is the stronger oxidizing agent. As reducing agents: $Zn > Cu > Ag$; As oxidizing agents: $Ag^+ > Cu^{++} > Zn^{++}$.

3. Test a strip of brightened lead in 5 ml of zinc nitrate solution and a second strip in 5 ml of copper(II) nitrate solution. Similarly test zinc and copper in separate solutions of lead(II) nitrate. In which of these four combinations do reactions occur? Write the balanced electronic equations for both oxidation and reduction actions where reactions occur. Also write ionic equations for the net reactions. How does lead compare with zinc and copper as a reducing agent? How does the Pb^{++} ion compare with the Zn^{++} ion and Cu^{++} ions as an oxidizing agent? Arrange the 4 metals tested in order of their relative strengths as reducing agents, placing the strongest first. Arrange the 4 metallic ions in order of their relative strengths as oxidizing agents, placing the weakest first.

Observations: Pb replaces Cu; Zn replaces Pb. $Pb - 2e^- \rightarrow Pb^{++}$; $Cu^{++} + 2e^- \rightarrow Cu$; $Pb + Cu^{++} \rightarrow Pb^{++} + Cu$ $Zn - 2e^- \rightarrow Zn^{++}$; $Pb^{++} + 2e^- \rightarrow Pb$; $Zn + Pb^{++} \rightarrow Zn^{++} + Pb$. Pb is a stronger reducing agent than Cu but weaker than Zn. Pb^{++} is a weaker oxidizing agent than Cu^{++} but stronger than Zn^{++}.
$Zn > Pb > Cu > Ag$; $Zn^{++} < Pb^{++} < Cu^{++} < Ag^+$.

4. Note the color of a 5 ml solution of iron(III) chloride solution. Add tin(II) chloride solution, dropwise near the end, until the color of the iron(II) chloride has disappeared. Describe the color change. The Fe^{+3} ion has been reduced to the Fe^{++} ion. What was the reducing agent? What change did it undergo? Write the balanced electronic equations and the ionic equation for the net reaction.

Observations: The color changes from yellow-brown to practically colorless (pale green). The reducing agent was the Sn^{++} ion. It was oxidized to Sn^{-4}.

$Fe^{+++} + e^- \rightarrow Fe^{++}$; $Sn^{++} - 2e^- \rightarrow Sn^{++++}$; $2\,Fe^{+++} + Sn^{++} \rightarrow 2Fe^{++} + Sn^{++++}$

5. Divide 2 g of oil-free iron filings into two approximately equal portions. Save one portion for Part 6, and to the other portion (test tube), add 4 ml of water and then 2 ml of concentrated hydrochloric acid. Warm (do not boil) the mixture until a moderate reaction is under way. What gas is evolved? Allow to stand until the solution is clear. Recall the colors of iron(III) chloride and iron(II) chloride solutions in Part 4. Which iron ion is produced in this reaction? What is reduced? Write the balanced electronic equations for both actions and the ionic equation for the net reaction. Which is the stronger reducing agent, Fe or H_2?

Observations: H_2 is evolved. The almost colorless Fe^{++} ion is produced. The Fe is oxidized and the hydronium ion is reduced.

$Fe - 2e^- \rightarrow Fe^{++}$; $2\,H_3O^+ + 2e^- \rightarrow 2H_2O + H_2(g)$;

$Fe + 2H_3O^+ \rightarrow Fe^{++} + 2H_2O + H_2(g)$.

Fe is the stronger reducing agent.

6. To 5 ml of iron(III) chloride solution (test tube), add 1 ml of concentrated hydrochloric acid and the second portion of iron filings retained from Part 5. Describe the action. Recall your previous comparison of the colors of iron(III) chloride and iron(II) chloride solutions. What reduction occurs? H_2 is the reducing agent. What is the purpose of the iron filings and hydrochloric acid? Write the pair of balanced electronic equations. Based on the reactions of Part 5 and Part 6, list H_2, Fe, and Fe^{++} in the order of their relative strengths as reducing agents.

Observations: H_2 gas is evolved, the color changes from yellow-brown to almost colorless (pale green). The Fe^{+++} is reduced. The iron filings and HCl yield the H_2 which is the reducing agent.

$Fe^{++} + e^- \rightarrow Fe^{++}$; $H_2 - 2e^- \rightarrow 2H^+$;

$2Fe^{+3} + H_2 \rightarrow 2Fe^{++} + 2H^+$

7. Dissolve 1 or 2 small crystals of potassium permanganate in 10 ml of water. Note the intensity of the color. Recall the color of common potassium salts. Which ion of the salt is responsible for this color? Dissolve 2 or 3 small crystals of iron(II) sulfate in 5 ml of water. Acidify with 2 or 3 drops of concentrated sulfuric acid. Now add the potassium permanganate solution to the solution containing the Fe^{++} ions, a very little at a time (mix) and finally dropwise, until the color remains. The permanganate, MnO_4^-, ion is a strong oxidizing agent. The manganese(II), Mn^{++}, ion is practically colorless. What occurred during the addition of the permanganate solution? What was oxidized? What was reduced? What was indicated by the return of the permanganate color to the solution? What changes in oxidation number occurred?

Observations: The permanganate ion is responsible for the color. The almost colorless (pale green) solution of Fe^{++} changes to the yellow-brown of Fe^{+3}. The Fe^{++} is oxidized to Fe^{+3} and the permanganate ion is reduced to Mn^{++}. When the purple color of the MnO_4^- ion returned (after oxidation of Fe^{++} to Fe^{+3}) the permanganate ion was in excess. The Mn changed its oxidation number from +7 to +2, the Fe changed its oxidation number from + 3 to + 2.

EQUATIONS

1. $Zn + Cu(NO_3)_2 \rightarrow Zn(NO_3)_2 + Cu(s)$
2. $Fe + 2\,HCl \rightarrow FeCl_2 + H_2(g)$
3. $2\,FeCl_3 + SnCl_2 \rightarrow 2\,FeCl_2 + SnCl_4$
4. $2\,FeCl_3 + H_2 \rightarrow 2\,FeCl_2 + 2\,HCl$
5. $10\,FeSO_4 + 2\,KMnO_4 + 8\,H_2SO_4 \rightarrow 5\,Fe_2(SO_4)_3 + 2\,MnSO_4 + K_2SO_4 + 8\,H_2O$

Experiment 42

NAME ..... PERIOD ..... DATE .....

INSTRUCTOR'S COMMENTS .....

Oxidation-Reduction Titration

PURPOSE: To determine a percentage composition by means of an oxidation-reduction titration.

APPARATUS: Balance, sensitive to 0.01 g; 3 beakers, 150 ml; buret, preferably with glass stopcock; clamp, buret; graduated cylinder; ring stand; vial, shell, small size (litmus paper vial will do); wash bottle.

MATERIAL: Unknown iron(II) salt; sulfuric acid, dilute (1:6); standard solution of potassium permanganate (approximately 0.02 *M*).

INTRODUCTION: Any reaction in which there is a distinct change in some characteristic of the solution when equivalent quantities of the reactants are present may lend itself to the titration technique. Certain exidation-reduction reactions are accompanied by a distinct color change when equivalent quantities of the oxidizing ion and reducing ion are present in the solution. The iron(II) ion, Fe^{++}, is oxidized to iron(III) ion, Fe^{-3}, by the permanganate ion, MnO_4^-, when in acid solution. The MnO_4^- ion is reduced to the manganese(II) ion, Mn^{++}, in the reaction. Of these four ions, only the permanganate is intensely colored. Thus as long as Fe^{++} ions are present in a solution to which MnO_4 ions are being added, the MnO_4^- color will disappear as Mn^{++} ions are formed. When all Fe^{++} ions have been oxidized, MnO_4^+ ions will no longer be reduced to Mn^{++} and the characteristic color will reappear. Thus the first faint appearance of the pink color of the MnO_4^- ion indicates the *end point* of the titration.

In the case of oxidizing and reducing agents, the particular oxidation-reduction reaction must be known in order to express the gram-equivalent weights of the reactants. *In a given oxidation-reduction reaction, the gram-equivalent weight of the oxidizing agent or the reducing agent is equal to the gram-formula weight divided by the number of electrons lost or gained.* For example, in Part 4 of Experiment 41, the iron(III) ion, Fe^{+++}, was reduced to Fe^{++}. Tin(II) ion, Sn^{++}, was the reducing agent, being oxidized in the process to Sn^{++++}. The electronic equations for this reaction show the number of electrons gained or lost.

$$2Fe^{+++} + 2e^- \rightarrow 2Fe^{++}$$
$$Sn^{++} - 2e^- \rightarrow Sn^{++++}$$

The gram-equivalent weights of the reactants in this reaction are:

$$\text{g-eq. wt. } Fe^{+++} = \frac{2Fe^{+++}}{e^- \text{ gained}} = \frac{2 \times 56\text{ g}}{2} = 56\text{ g}$$

$$\text{g-eq. wt. } Sn^{++} = \frac{Sn^{++}}{e^- \text{ lost}} = \frac{119\text{ g}}{2} = 59.5\text{ g}$$

In this oxidation-reduction titration, you will titrate a solution of a permanganate of known concentration into a solution of a weighed sample of an unknown iron(II) salt. By computing the equivalent weights of the two reactant ions for this particular reaction, and knowing the volume and concentration of the MnO_4^- solution and the mass of Fe^{++} salt, you will be able to determine the percentage composition of iron in the unknown salt.

PROCEDURE: **1.** Place about 1 g of an unknown iron(II) salt in a small vial (a watch glass will serve) and weigh accurately to 0.01 g. Record this mass in the Data Table. Now pour approximately half of the salt into a clean dry beaker labeled *No. 1.* Reweigh the vial and remaining salt. Record. Pour this portion of the salt into a second clean beaker labeled *No. 2.* Reweigh the empty vial and record.

2. Rinse a clean buret, first with a few milliliters of distilled water, and then with a few milliliters of the standard solution of potassium permanganate. Mount the buret in position and fill with the permanganate solution. Draw off enough of the solution to remove the air from the jet tip and record the initial reading.

3. Add about 50 ml of distilled water to the No. 1 beaker and dissolve the iron(II) salt. Add 5 ml of dilute sulfuric acid and titrate immediately until a final drop of the permanganate solution causes a pink coloration to remain. This may easily be detected if the beaker is resting on white paper and a beaker of distilled water is placed alongside for comparison. Record the final buret reading.

4. Prepare the No. 2 sample and titrate as in Part 3. Unless the buret required refilling before commencing the second titration, the initial reading for this second trial will be the final reading for the first trial.

DATA TABLE

Student answers.

First mass of vial and salt		g
Second mass of vial and salt		g
Mass of empty vial		g
	No. 1	No. 2
Mass of sample of Fe^{++} salt	g	g
Initial buret reading	ml	ml
Final buret reading	ml	ml
Volume of MnO_4^- solution used	ml	ml
Mass of MnO_4^- reduced (computed)	g	g
Mass of Fe^{++} oxidized (computed)	g	g
Percentage composition of iron as Fe^{++} in salt	%	%
	Average value	%

CALCULATIONS: *Show all computations in smooth form on a separate sheet of paper.*

1. As a result of the oxidation-reduction action, the oxidation state of iron was raised from +2 to +3. Determine the change in the oxidation state of manganese by the reduction of MnO^{-4} to Mn^{++}. Write the balanced electronic equations for both oxidation and reduction systems.

2. Refer to the explanation in the Introduction if necessary, and then calculate the g-eq. wt. of Fe^{++} and the g-eq. wt. of MnO_4^- for this particular oxidation-reduction reaction.

g-eq. wt. Fe^{++} = g.

g-eq. wt. MnO_4^- = g.

3. From the volume of MnO_4^- solution used with each sample and the known concentration, determine the mass of MnO_4^- used with each sample.

4. Now knowing the g-eq. wt. of Fe^{++} and the g-eq. wt. of MnO_4^- for this reaction and the actual mass of MnO_4^- reduced by each sample of Fe^{++} salt, compute the actual mass of Fe^{++} oxidized in each sample.

5. From the mass of salt and the mass of Fe^{++} oxidized in each sample, compute the percentage composition of iron as Fe^{++} in this salt for both samples. What is the average value of your two percentage-composition determinations? Record the results of all computations in the Data Table.

Experiment 43(a)

NAME ..... PERIOD ..... DATE .....

INSTRUCTOR'S COMMENTS .....

Electrochemical Cells, Half-Cell Reactions

PURPOSE: To construct several electrochemical cells and study their performance.

APPARATUS: Clips, alligator; copper wire, insulated, No. 18; demonstration cell (a glass tumbler, porous cup, battery top); electrodes, carbon; glass bends (U-shaped for salt bridge); graduated cylinder (10 ml); medicine dropper; voltmeter, d-c, 0-1.5 v.

MATERIAL: Litmus paper, red; sandpaper, 1 × 5 cm; ammonia-water solution (6.0 *M*); hydrochloric acid, concentrated; solutions of: bromine water; chlorine water; copper(II) sulfate (1.0 *M*); iodine (aqueous); iron(II) chloride (Appendix); iron(III) chloride (0.1 *M*); potassium iodide (0.1 *M*) plus starch (Appendix); potassium permanganate (1.0 *M*); zinc sulfate (1.0 *M*).

INTRODUCTION: In oxidation-reduction reactions, electrons are transferred from the substance oxidized to the substance reduced. Oxidation-reduction reactions that occur spontaneously convert chemical energy into heat when the chemicals react. The reactants may be so arranged that the electron transfer will occur through a conducting wire connected between them. In this arrangement, chemical energy is converted to electric energy. Such an arrangement consists of two half-cells connected by a porous partition, or a conducting salt bridge. It is called an electrochemical cell.

In the first part of this experiment, you will construct an electrochemical cell (Daniell Cell) capable of producing an electromotive force of approximately 1 volt. To understand and interpret the performance of this cell and the others which you construct, you will need to understand the oxidation-reduction reaction which occurs in each case. Refer to the Table of Standard Electrode Potentials (Electromotive or Activity Series) in Chapter 21, Section 21.11, MODERN CHEMISTRY (1974) for specific half-cell reactions and oxidation potentials.

SUGGESTION: Students may work in pairs and different pairs may be assigned to construct and study the performance of one or more of the electrochemical cells. Toward the end of the period, the results obtained by various pairs may be reported to the class and compared with results that would be obtained by the theoretical use of the Table of Standard Electrode Potentials.

PROCEDURE: 1. *Electrochemical cell.* Construct a Daniell Cell, using a small demonstration cell consisting of a glass tumbler, an unglazed porcelain cup, and a battery top. Fill the porous cup to within 1 cm of the top with 1.0-*M* zinc sulfate solution. Place the cup in the glass tumbler and then fill the tumbler with 1.0-*M* copper (II) sulfate solution to the same level as that of the zinc sulfate solution. Brighten a copper electrode and a zinc electrode with sandpaper and clamp them into position in the battery top. Place the battery top on the tumbler so the copper electrode is suspended in copper(II) sulfate solution and the zinc electrode is suspended in zinc sulfate solution.

Connect one terminal of a d-c voltmeter to one electrode terminal on the battery top by a convenient length of copper wire. Attach another section of wire to the other voltmeter terminal. Momentarily touch the free end of this wire to the other electrode terminal on the battery top, observing the meter needle for deflection. If there is no deflection, reverse the connections to the battery top. Result? What voltage does the meter register?

The meter terminals are marked "+" and "—" to identify the anode and cathode of the electrochemical cell. Which metal is acting as the cathode? Which is the anode? Trace the path of electrons through the external circuit. At which electrode is reduction occurring? At which electrode is oxidation occurring? Consult the Table of Standard Electrode Potentials mentioned in the Introduction. Does this table support your conclusions?

Disconnect your cell, rinse the two electrodes, and return the meter. Remove the porous cup from the copper(II) sulfate solution. Rinse both containers thoroughly.

Observations: Should read 1.1 volts. Zn acts as cathode, Cu as the anode. The electrons flow from the Zn

to the Cu through the external circuit. Reduction is occurring at the anode(Cu) and oxidation at the cathode (Zn). The Table of Oxidation Potentials supports the conclusions.

2. *Identification tests.* These preliminary tests are to be performed so that they may be used later to identify some of the products which result from the operation of electrochemical cells. They are arranged in sections to correspond to the various electrochemical cells to be set up according to Part 3. The assigned set(s) of electrochemical cell(s) should be set up and allowed to operate first according to Part 3. During this time interval, you will proceed according to Part 2.

a. For KI and $FeCl_3$ cell: Add 5 drops of ammonia-water solution to 5 ml samples of solutions of iron(II) chloride and iron(III) chloride respectively. Note the color of the precipitates formed. These will identify the respective ions. Add 2 ml of starch colloidal suspension to 5 ml of the aqueous solution of iodine. Note the resultant color. To 5 ml of iron(III) chloride add 5 ml of the solution containing potassium iodide and starch. Result? What is one identifiable product of this oxidation-reduction reaction?

Observations: $Fe(OH)_3$ is a red-brown precipitate while $Fe(OH)_2$ is a white precipitate which turns green in presence of air. Starch and iodine solution produce a blue-black color. Addition of KI to $FeCl_3$ causes the starch to turn to blue-black indicating production of I_2.

b. For KI and Br_2 cell: Add 5 drops of bromine water to 5 ml of the mixture of potassium iodide and starch. Result? What is one identifiable product of this oxidation-reduction reaction?

Observations: Bromine water addition causes the starch to turn blue-black as the potassium iodide is changed to I_2. Therefore I_2 is identifiable.

c. Put one drop of chlorine water on a piece of red litmus paper. Result? Carefully test for the odor of chlorine in chlorine water by using the wafting motion described in Fig. F-5. Add 5 ml of the potassium permanganate solution to 1 ml of hydrochloric acid. Test for the odor of a product of this oxidation-reduction reaction by performing the odor test. Insert a piece of red litmus paper into the test tube. Result? What happened to the color of the permanganate solution?

Observations: Chlorine water changes red litmus paper to white. $KMnO_4$ and HCl produce chlorine which is identified by its suffocating odor and bleaching action on red litmus paper. The purple permanganate color changes to colorless gradually fading through a pink to colorless Mn^{++}.

3. *General.* For the electrochemical cell(s) assigned to you, pour 5 cm of each of the solutions into separate test tubes. Insert the salt bridge and the carbon electrodes (Fig. 43-1). Attach one end of the wire from a carbon electrode to the one terminal of the d-c voltmeter. Momentarily touch the free end of the wire from the other electrode to the other voltmeter terminal. If there is no deflection, reverse the connections to the voltmeter. If there is a deflection, make a permanent connection. Record the observed voltage in the Data Table. Also record the solution in which solution the electrode is anode. After making the reading, disconnect the wires from the voltmeter and twist them together. Permit the reaction to proceed in the connected half-cells for 10 minutes. Record the results of the identification tests which are described

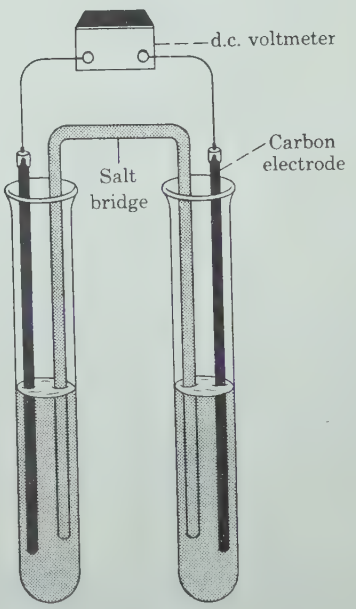


Fig. 43-1

DATA TABLE

Half-cell reactants	Identification of inserted electrode	Half-cell reaction equation	Observed voltmeter reading	Potential difference (Table of Standard Electrode Potentials)
A. KI	cathode	$2I^- \rightarrow I_2 + 2e^-$	student answer	−0.54v
$FeCl_3$	anode	$Fe^{+++} + e^- \rightarrow Fe^{++}$	student answer	+0.77v +0.22v
B. KI	cathode	$2I^- \rightarrow I_2 + 2e^-$	student answer	−0.54v
Br_2	anode	$Br_2 + 2e^- \rightarrow 2Br^-$	student answer	+1.06v +0.52v
C. $KMnO_4$	anode	$MnO_4 + 8H^+ + 5e^- \rightarrow 4H_2O + Mn^{++}$	student answer	+1.49v
HCl	cathode	$2Cl^- - 2e^- \rightarrow Cl_2$	student answer	−1.36v +0.13v

in the following sections. At the conclusion of the experiment, remove the salt bridge, disconnect the twisted wires, and rinse the test tubes and electrodes thoroughly.

a. For KI and $FeCl_3$ cell: What change do you note in the test tube containing the potassium iodide-starch mixture? What is one product in this half-cell? If your experiment has proceeded to the point where the contents of the tube containing iron(III) have become almost colorless, perform the test with ammonia-water solution. What is an identifiable product in this half-cell?

b. For KI and Br_2 cell: What change do you note in the test tube containing the potassium iodide-starch mixture? What is one product of this half-cell reaction?

c. For $KMnO_4$ and HCl cell: Note any change in the color of the potassium permanganate solution. Perform the odor test for the test tube containing the hydrochloric acid. Insert a piece of red litmus paper and note the result. What is one product of this half-cell reaction?

Observations: a. The KI-starch mixture turns blue-black indicating the production of I_2. If the $FeCl_3$ half-cell has become almost colorless, ammonia-water addition yields the white to green $Fe(OH)_2$ precipitate identifying the Fe^{++} as a product.

b. The KI-starch mixture turns blue-black indicating and identifying I_2 as a product.

c. The permanganate solution turns pink and may turn colorless indicating change to Mn^{++}. The odor of Cl_2 comes from the HCl tube and inserted red litmus paper is bleached. Cl_2 is a product.

EQUATIONS: Write complete balanced equations for these oxidation-reduction reactions.

$2KI + 2FeCl_3 \rightarrow 2FeCl_2 + I_2$

$2KI + Br_2 \rightarrow 2KBr + I_2$

$2KMnO_4 + 16HCl \rightarrow 8H_2O + 2KCl + 2MnCl_2 + 5Cl_2$

Experiment

43 (b) Electrolytic Cells

NAME . . . . . PERIOD . . . . . DATE . . . . . .

INSTRUCTOR'S COMMENTS . . . . . .

PURPOSE: To construct electrolytic cells cells and study their performance.

APPARATUS: Beaker, 50 ml; copper wire, insulated No. 18; demonstration cell (a glass tumbler, porous cup, battery top); 3 dry cells; electrodes, 2 Pt; pipet, 10 ml; watch glasses.

MATERIAL: Litmus paper, red and blue; solutions of: copper(II) sulfate (0.5 *M*), phenolphthalein indicator; potassium iodide (0.1 *M*), sodium sulfate (0.2 *M*).

SUGGESTIONS: If a sufficient supply of platinum electrodes is unavailable, students should be encouraged to construct equivalent apparatus using carbon electrodes. Results for Parts 2 and 3 of this experiment may be duplicated by the use of such apparatus. A blue solution of copper(II) bromide may be used instead of copper(II) sulfate for Part 2. The test for bromine, using carbon tetrachloride, may be used for the anode product. Part 1 may be performed in a specially constructed apparatus briefly described under Further Experimentation.

PROCEDURE: 1. Assemble the demonstration cell with 0.2 *M* sodium sulfate solution in both the tumbler and the porous cup. Mount the two platinum electrodes in the battery top, one extending into the solution in the cup and the other into the solution outside the cup. Connect three dry cells *in series* as a battery, using two short lengths of copper wire. Connect the negative terminal of the battery to the platinum electrode which extends into the cup. Connect the positive terminal of the battery to the other electrode. See Fig. 43-2.

Observe the action at each electrode. Describe it. Recall the action at the anode and at the cathode during the electrolysis of water. What ion was discharged at the cathode? What ion was discharged at the anode, and what gas was evolved? Does this action appear to be similar? Would you expect it to be? What two positive ions in the solution are attracted to the cathode? Which would you predict is more easily discharged? Evidence? Do you find agreement at the Table of Standard Electrode Potentials?

Allow the electrolytic cell to operate for at least 5 minutes. Pipet about 2 ml of solution from the porous cup onto a watch glass resting on a sheet of white paper. Add a drop of phenolphthalein. Result? Rinse the pipet and similarly test a 2 ml portion of the solution outside the porous cup, using a second watch glass. Result? Explain. Using a clean stirring rod, test the reaction of each of the cell solutions with red and blue litmus paper. Result? Explain. Discard the solution and rinse the electrodes and the two containers thoroughly, but keep the electrode assembly intact.

Observations: A gas is being liberated at each electrode. The solution from inside the porous cup turns phenolphthalein pink indicating production of OH^- ion. The solution from outside the cup does not change phenolphthalein. However this solution does turn blue litmus to red indicating production of hydronium ion while the cell solution inside the cup (cathode) confirms production of OH^- by turning red litmus to blue.

2. Pour the copper(II) sulfate solution retained from Part 1 in the glass tumbler (do not use the porous cup). Insert the two platinum electrodes and allow the electric current to pass for about 1 minute. Observe the action at both electrodes. Is a gas evolved at each? Life out the electrodes and examine them. Result? What two positive ions in the solution are attracted to

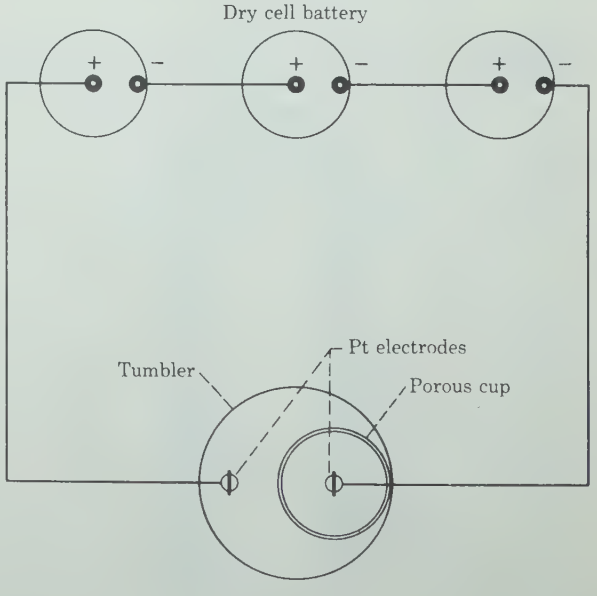


Fig. 43-2

the cathode? Which has been discharged? Is this observation supported by the Table of Standard Electrode Potentials?

Remove the platinum *anode* and substitute in its place a copper electrode. From the action you have observed, determine how you reconnect the electrodes to the battery to remove the plating from the remaining platinum electrode. Try it. Result? You can quickly restore the platinum electrode to its original condition. Explain.

Observations: A gas is liberated at the anode while a copper deposit forms on the cathode. Both hydronium and copper(II) ions were attracted to the cathode but the copper(II) ions were discharged. This is supported by the Table of Standard Oxidation Potentials since the oxidation potential of the $Cu \rightleftarrows Cu^{++} + 2e^-$ half-reaction is lower than that $H_2 + 2H_2O \rightleftarrows 2H_3O^+ + 2e^-$ half-reaction.

3. Pour about 10 ml of 0.1-*M* potassium iodide solution in a 50-ml beaker. Add 3 drops of phenolphthalein indicator. Connect the battery to the two platinum electrodes (removed from the battery top) and lower them into the solution at opposite sides of the beaker. Leave them in the KI solution just long enough to observe a distinct action at each electrode. Is there a gas evolved at either electrode? What color changes occur? How do you explain the color change at the anode? Is this the result of oxidation or reduction? Which ion is discharged more easily at the cathode, the K^+ or the H_3O^+? Evidence? Account for the color change at the cathode. Does the anode appear to be plated? Rinse it in a little KI solution. Result? Rinse both electrodes carefully in water.

Observations: A gas is evolved at the cathode around which the phenolphthalein changes to pink. The solution around the anode changes from colorless to brown due to oxidation of the I-ion to I_2. The hydronium ion is more easily discharged than the K^+ ion although the more modern explanation is that water is reduced at the cathode producing H_2 and OH^- ion. The anode may appear to have a brown deposit of I_2 which is easily removed by rinsing in KI solution.

QUESTIONS: Explain the following briefly but concisely. Write the electrode reactions and the net equation for the overall electrolysis in each case.

1. The oxidation-reduction action in the Pt-Pt, sodium sulfate electrochemical cell. . . .

Cathode: $4\ H_2O + 4e \rightarrow 4\ OH^- + 2\ H_2(g)$

Anode: $6\ H_2O - 4e^- \rightarrow 4\ H_3O^- + O_2(g)$

$4\ H_3O^+ + 4\ OH^- \rightarrow 8\ H_2O$

Net: $2\ H_2O \rightarrow 2\ H_2(g) + O_2(g)$

2. The oxidation-reduction action in the Pt-Pt, copper(II) sulfate electrolytic cell. . . .

Cathode: $Cu^{++} + 2e^- \rightarrow Cu$

Anode: $4\ OH^- - 4e^- \rightarrow 2\ H_2O + O_2(g)$

Net: $2\ Cu^{++} + 4\ OH^- \rightarrow 2\ Cu + 2\ H_2O + O_2(g)$

3. The different color changes at the electrodes in the Pt-Pt, KI electrolytic cell. . . .

Cathode: $2\ H_3O^+ + 2\ e^- \rightarrow 2\ H_2O + H_2(g)$

Anode: $2I^- - 2e^- \rightarrow I_2$

Net: $2\ H_3O + 2I^- \rightarrow 2\ H_2O + I_2 + H_2(g)$

4. How does the Table of Standard Electrode Potentials enable you to predict which of the positive ions present at the cathode of an electrolytic cell will be discharged?

The ion which is the stronger oxidizing agent is the one which undergoes reduction (discharge).

FURTHER EXPERIMENTATION: The electrolysis of aqueous sodium sulfate may be explained as actual reduction and actual oxidation of water rather than as reduction of hydronium ions and oxidation of hydroxide ions. This viewpoint is an acceptable explanation since the concentrations of these ions in the neutral sodium sulfate solution is 1×10^{-7} *M*. It is consistent with the reduction of water by reducing agents such as metallic sodium and its oxidation by chlorine. The reactions are

Reduction $2H_2O + 2e \rightarrow H_2\,(g) + 2OH^-$
Oxidation $6H_2O \rightarrow O_2\,(g) + 4H_3O^+ + 4e^-$

Balancing these half reactions according to the law of conservation of electrons yields the equation

$$10H_2O \rightarrow O_2\,(g) + 4H_3O^+ + 4OH^- + 2H_2\,(g)$$

which produces the net equation

$$2H_2O \rightarrow 2H_2\,(g) + O_2\,(g)$$

The apparatus is constructed from a plastic dish according to the directions described in the *Journal of Chemical Education*, 34, 291 (1957), Teichman, Louis. It is also described in a reprint appearing in *Selected Readings in General Chemistry*, Keiffer, Wm. F.; Fitzgerald, Robt. K.; Chemical Education Publishing Co., Easton, Pennsylvania. The electrodes should be made from stainless steel rods and should be approximately 7 cm long. The dish is filled with the solution. Two test tubes are filled by sinking them in the solution and a piece of red and a piece of blue litmus paper is inserted into each test tube. The tubes are lifted to the vertical position and placed over the respective electrodes. As electrolysis proceeds, the litmus papers change indicating the production of hydronium and hydroxide ions. The gaseous products are identified by use of a glowing and a flaming splint. Before these tests are performed, the open ends of the tubes are stoppered under the solution for convenient removal. After the tests for oxygen and hydrogen are performed, the liquid contents of each test tube are poured back into the plastic dish, stirred, and litmus paper tests are again performed with the resultant mixture. Litmus paper remains unchanged indicating the stoichiometric production of hydronium and hydroxide ions. The apparatus may then be used by another student without any change of the solution.

Experiment 44

NAME . . . . . PERIOD . . . . . DATE . . . . . .

INSTRUCTOR'S COMMENTS . . . . . .

Elements of Period Three

PURPOSE: To study the properties of the elements of Period Three and some of their representative compounds.

APPARATUS: Balance, platform; beaker, 150 ml; 2 bottles, wide mouth; burner and tubing; cardboard; clamp, test tube; forceps; 2 glass plates; graduated cylinder; molecular model set (Sargent); scissors; Scotch tape; spoon, deflagrating; test tubes; tongs.

MATERIAL: Ammonium chloride; iron(II) sulfide; magnesium ribbon; phosphorus, red; sodium chloride; sodium peroxide; sulfur, roll; asbestos paper; Hydrion papers (A, B, D); litmus papers, red and blue; wooden splints; ammonia-water, dilute (1:4); hydrochloric acid, dilute (1:4); sulfuric acid, dilute (1:6); solutions of aluminum chloride (0.2 *M*); sodium hydroxide (2.5 *M*).

INTRODUCTION: The elements of Period Three illustrate the variation in properties which occurs as one goes across a period. In this period, the first element is a highly reactive metal. Then come other metals of decreasing activity. These are followed by a metalloid and then by nonmetals of increasing activity. The last element in a period is a noble gas. The properties of representative hydroxides, oxides, and binary hydrogen compounds illustrate the variations in the fundamental natures of the elements of the period.

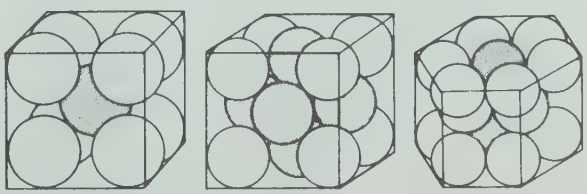
PROCEDURE: 1. Assemble models of the crystals representative of the metals sodium, magnesium, and aluminum according to Fig. 44-1. Use the yellow spheres in the molecular model set. Nine spheres are required for the model of the sodium crystal. The construction of the magnesium crystal requires 17 spheres, while that of the aluminum crystal requires 14 spheres. Construct cardboard retainers of suitable size (approximately 8 cm in height) using tape to hold them together. The arrangements (packing) of the spheres in the cases of magnesium and aluminum represent the closest possible arrangements of identical spheres (atoms; ions). That of sodium represents a less tightly packed arrangement. Refer to Chapter 22, MODERN CHEMISTRY, 1974, and state the three physical properties of sodium which may be related to the crystal arrangement.

Observations: Sodium has a lower melting point, is soft enough to be cut with a knife, and has a much lower density due to looser crystal arrangement.

2. Construct a model of the silicon crystal according to Fig. 31-1 in Experiment 31. What is the electron configuration of the silicon atom? What type of hybridization produces this crystal type? Explain the great difference in melting points that occurs between aluminum and silicon in terms of the change in crystal structure which occurs.

Observations: The configuration of the Si atom (ground state) is $1s^2 2s^2 2p^6 3s^2 3p^2$. The type of hybridization producing the macromolecular cubic structure is sp^3 hybridization. The change from the metallic to the macromolecular structure accounts for the great difference in melting points between the two consecutive elements Al and Si.

3. Construct models of the molecules of phosphorus, sulfur, and chlorine (see Fig. 44-2) according to the following directions: phosphorus (4 light blue spheres); sulfur (8 black spheres); chlorine (2 green spheres). How do the magnitudes of the melting and boiling points of these three elements compare with those of the first four elements of the period? Explain these in terms of the forces that hold these elements together in the solid state.



Sodium Aluminum Magnesium

Fig. 44-1

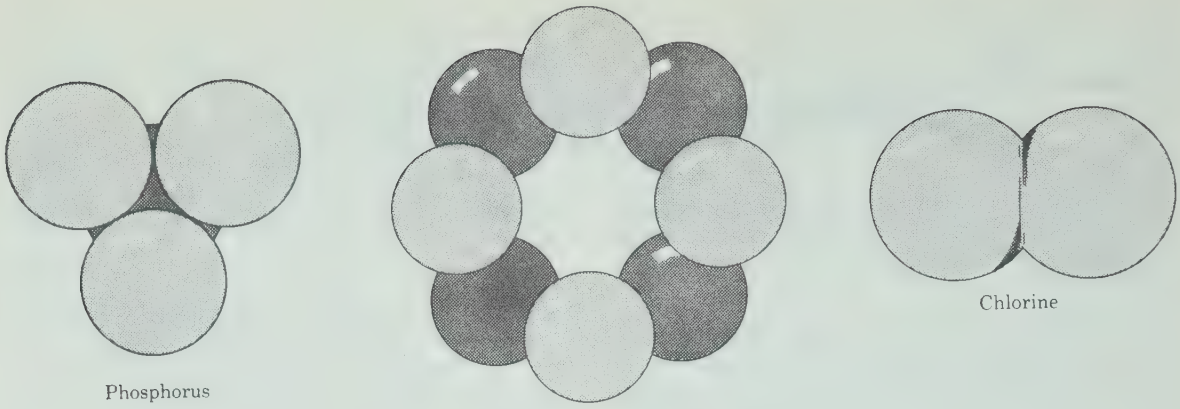


Fig. 44-2

Observations: These elements have much lower melting points than those of the first four elements of this period. Chlorine is a gas at room temperature. In each case the molecules of these non-metals are held together by weak van der Waals forces.

4. *a.* Add 5 ml of water to 1 g of sodium peroxide. Use a glowing splint to identify the gas produced. Test the contents of the test tube with red and blue litmus paper. Results? Test with Hydrion paper (range 11-13). Result? Approximate pH?

b. Hold one end of a 5-cm piece of magnesium ribbon with the tongs and ignite the other end in the burner flame. Hold the burning magnesium over a beaker so that the product drops into the beaker. Add 5 ml of water to the product and heat gently. Test the contents of the beaker with red and blue litmus paper. Result? Test with Hydrion paper (range 11-13). Result? Approximate pH? Compare pH for *a* and *b*.

c. Put a piece of sulfur, the size of a small pea, in the asbestor-lined bowl of the deflagrating spoon, ignite it in the burner flame, and lower it into a bottle of air. Add 10 ml of water to the bottle contents, cover with a glass plate, and shake the contents. Add a piece of red litmus paper and one of blue to the contents of the bottle. Result?

d. Repeat *c* using an amount of red phosphorus equal to the bulk of a match head. Result? What do the results of Procedures *a* through *d* indicate about the reactions of the oxides of the respective elements with water?

Observations: a. Oxygen is produced. The test tube contents turn red litmus to blue and Hydrion paper indicates a pH of 13. These tests indicate production of NaOH as second product.

b. The heated MgO and H_2O change red litmus to blue and approximate pH by Hydrion paper is about 11. Comparison of the pH with that of a. (above) indicates that $Mg(OH)_2$ is a much weaker base than NaOH.

c. Blue litmus paper turns red indicating the production of acid: hydronium ion.

d. Phosphorus burns vigorously and the solution of the oxide produced turns blue litmus to red indicating production of acid: hydronium ion. The oxides of Na and Mg produce bases, while the oxides of the nonmetals S and P produce acids.

5. To 5 ml of aluminum chloride solution, add 1 ml of ammonia-water solution. Result? What product is formed? The gelatinous precipitate formed does not settle readily and may be regarded as a suspension. Transfer half of this suspension to a second test tube. To the suspension in one test tube, add 1 ml of sodium hydroxide solution. To the suspension in the other test tube add 1 ml of dilute hydrochloric acid. Compare the results. What property of aluminum hydroxide is thus illustrated?

Observations: A gelatinous precipitate of $Al(OH)_3$ is produced. The $Al(OH)_3$ dissolves both in HCL and NaOH solutions. This indicates that $Al(OH)_3$ is amphiprotic.

6. For each reaction described in this part of the experiment, add 2 ml of liquid reagent to 1 g of solid reagent in a test tube held with the test tube clamp. Heat the test

tube gently. Then insert a piece of moistened red litmus paper and one of blue litmus paper into the vapor in the upper part of the test tube and record the results. Since the binary hydrogen compound of phosphorus is poisonous and flammable, that of nitrogen, the corresponding element in Period Two, will be substituted to show the variations in the properties of the binary hydrogen compounds of the non-metallic elements.

a. Add 2 ml of sulfuric acid to 1 g of sodium chloride and heat gently. What does the litmus paper reaction indicate? Insert a piece of moistened Hydrion paper (type A) into the vapors. Result? What is the approximate pH indicated?

b. Repeat Part *a* using 1 g of iron(II) sulfide with 2 ml of sulfuric acid. Results? What do the results of the Hydrion paper tests indicate about the relative strengths of the water solutions of the respective binary hydrogen compounds of chlorine and sulfur?

c. Add 2 ml of sodium hydroxide solution to 1 g of ammonium chloride. Heat gently and test the vapor produced with pieces of moistened red and blue litmus papers, as well as with Hydrion paper (Type A). Results? How would you expect the water solution of the binary hydrogen compounds of phosphorus to compare in acidity with those of chlorine and sulfur?

Observations: a. Litmus paper reaction: blue to red indicates production of an acid, HCl. The approximate pH is about 1.

b. Litmus paper turns red, the rotten egg odor of H_2S being perceptible. The pH paper indicates that H_2S is a much weaker acid than HCl.

c. Moistened red litmus turns blue, pH paper indicates pH of about 11. The hydride of P would probably be much less acidic than that of S and Cl.

FURTHER EXPERIMENTATION: Have students test the pH of solutions of sulfuric acid (0.5 *M*) and phosphoric acid (0.33 *M*) with Hydrion paper to compare the strength of the comparable oxy-acids of sulfur and phosphorus.

EQUATIONS: Write complete balanced equations for these reactions.

1. Reactions for Part 4.

a. $2Na_2O_2 + 2H_2O \rightarrow 4NaOH + O_2(g)$

b. $2Mg + O_2 \rightarrow 2MgO$

$MgO^- + H_2O \rightarrow Mg(OH)_2$ or $MgO + H_2O \rightarrow Mg^{++} + 2OH^-$

c. $S + O_2 \rightarrow SO_2$

$H_2O + SO_2 \rightarrow H_2SO_3$ or $SO_2 + 2H_2O \rightarrow H_3O^- + HSO_3^-$

d. $P_4 + 5O_2 \rightarrow P_4O_{10}$

$P_4O_{10} + 6H_2O \rightarrow 4H_3PO_4$ or $P_4O_{10} + 10H_2O \rightarrow 4H_3O^+ + 4H_2PO_4^-$

2. Reactions for Part 5 (3 equations).

$AlCl_3 + 3NH_4OH \rightarrow 3NaCl + Al(OH)_3(s)$

$Al(OH)_3(s) + OH^- \rightarrow AlO_2^- + 2H_2O$

$Al(OH)_3(s) + 3H_3O^- \rightarrow Al(H_2O)_6^{+3}$

3. Reactions for Part 6 (2 equations for each section; the second equation should be ionic, showing the reaction of the binary hydrogen compound with water).

a. $NaCl + H_2SO_4 \rightarrow NaHSO_4 + HCl(g)$

$HCl + H_2O \rightarrow H_3O^- + Cl^-$

b. $FeS + H_2SO_4 \rightarrow FeSO_4 + H_2S(g)$

$H_2S + H_2O \rightleftarrows H_3O^- + HS^-$

c. $NH_4Cl + NaOH \rightarrow NaCl + H_2O + NH_3(g)$

$NH_3 + H_2O \rightleftarrows NH_4 + OH^-$

Experiment 45

NAME . . . . . PERIOD. . . . . DATE . . . . . .

INSTRUCTOR'S COMMENTS . . . . . .

Flame Tests

PURPOSE: To show how certain metals may be identified by the color they impart to a flame.

APPARATUS: Burner and tubing; 2 cobalt glass plates; test tubes; 5-cm length of No. 24 platinum wire, sealed in the end of a glass tube 10 cm long. If platinum wire is not available, nichrome wire (not as satisfactory as platinum) may be used. Nichrome wire should be held with forceps.

MATERIAL: Hydrochloric acid, dilute (1:4); sodium chloride; 0.5-*M* solutions in distilled water of the A.R. grade nitrates of barium, calcium, lithium, potassium, sodium, and strontium, and of sodium chloride; unknown solutions.

INTRODUCTION: If you wish to secure good results in this experiment, your test tubes must be *scrupulously* clean. Your test tubes may be thoroughly cleaned by using the cleaning solution described in the Teachers' Edition to EXERCISES AND EXPERIMENTS IN CHEMISTRY. (CAUTION: *This cleaning solution is extremely corrosive and must not be spilled!*) After the cleaning solution has been used, the test tubes may be rinsed thoroughly in tap water and then in distilled water.

SUGGESTION: Have students examine sodium lines produced in a spectroscope using a sodium vapor lamp and then a sodium flame source. This may be repeated with mixtures of sodium and potassium.

PROCEDURES: **1.** Clean a platinum wire by dipping it first into some dilute hydrochloric acid in a test tube and then holding it in the colorless flame of your burner. Repeat until the wire imparts no color to the flame. Pour 4 ml of sodium *nitrate* solution into a *clean* test tube, and dip the *tip* of the clean platinum wire into the solution, and then hold it in the flame. Observe the color of the flame just above the wire. Heat only the tip of the wire. If you heat the glass tube into which the wire is sealed, you will break the glass.

Clean the wire as before and then test a solution of sodium *chloride* in the same manner. Repeat the test, dipping the wire into a little *dry* sodium chloride.

Observations: Student answer. . . . . .

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2. Repeat Part 1, using in turn 4 ml of the solutions of the nitrates of lithium, strontium, calcium, barium, and potassium. Clean the wire thoroughly after each test. In the cases of lithium and strontium, observe which flame is more persistent and takes longer to burn off the wire. Also note the difference in the shades of color produced. When you have tested the calcium flame and then dipped the wire into hydrochloric acid and back into the flame when cleaning it, you often get an excellent flame of calcium momentarily. Record your observations in the accompanying table.

Observations: Students answer. . . . . .

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3. If two salts are present in the same solution, the color of one flame may obscure that of the other. Sometimes it is possible to absorb one color and not the other. Examine the sodium flame through at least two thicknesses of cobalt glass. Repeat, using the potassium flame with the cobalt glasses.

Flame test a mixture of the solutions of the nitrates of sodium and potassium with a clean wire. Observe the color the mixture imparts to the flame when viewed without the cobalt glasses. Repeat the test, but observe the flame as seen through the cobalt glasses. Explain any differences observed. Record your observations as before.

Observations: Student answer. . . . . .

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4. Secure an unknown solution from your instructor. Test it in the flame as in this experiment in order to identify the metallic ion present.

DATA TABLE Student answer.

Metal in compound	Color of flame
sodium	
lithium	
strontium	
calcium	
barium	
potassium	
sodium (cobalt glass)	
potassium (cobalt glass)	
sodium and potassium	
sodium and potassium (cobalt glass)	
unknown metal	

CONCLUSION: The unknown metal was Student answer.

QUESTIONS: Answer in complete statements.

1. Is the flame coloration a test for the metal or for the acid radical? It is a test for a metal

2. Why do dry sodium chloride and the solutions of sodium nitrate and sodium chloride all impart the same color to the flame? They all contain sodium.

3. Describe the test for sodium and potassium when both are present. Observe the flame directly. It will be yellow because of sodium. The violet color of the potassium flame is obscured. Next view through cobalt glass. The yellow color of sodium is absorbed, and the violet color of the potassium is visible.

4. How would you characterize the flame test with respect to its sensitivity? The test is extremely sensitive and very small amounts can be identified.

5. What difficulties may be encountered in the use of the flame test for identification? The yellow sodium may be found in most things because traces of sodium are often present as impurities. The shades of red produced by lithium and strontium are somewhat hard to distinguish. The calcium flame is not satisfactory partly because almost all calcium compounds usually contain sodium compounds as an impurity.

NAME . . . . . PERIOD . . . . . DATE . . . . . .
INSTRUCTOR'S COMMENTS . . . . . .

Experiment 46 Group II Metals and Their Compounds

PURPOSE: To study the comparative properties of some of the metals of Group II and their compounds.

APPARATUS: Balance, platform; bottle, wide mouth; burner and tubing; clamp, buret; funnel; funnel tube; glass bend; glass plate; graduated cylinder; 5 cm length of No. 24 platinum wire, sealed in the end of a glass tube (10 cm); ring stand; rubber stopper, 2-hole; test tubes, Pyrex; tubing, delivery.

MATERIAL: Barium sulfate; calcium, turnings; calcium sulfate, powder; magnesium, turnings; marble chips; filter papers; Hydrion papers (A, B, D); wooden splints; solutions of: barium hydroxide (saturated); barium chloride (0.5 *M*); calcium chloride (0.5*M*); calcium hydroxide (saturated); magnesium chloride (0.5 *M*).

INTRODUCTION: The alkaline earth metals, in general, resemble the alkali metals in their activity. They form oxides (or peroxides) and hydroxides with general similarities to those of the alkali metals. Their hydroxides, carbonates, and sulfates vary in solubility. The relative solubilities of the hydroxides explain their differences in alkalinity. The relative solubilities of their sulfates and carbonates are used in qualitative analysis in separating and identifying metallic ions.

PROCEDURE: 1. *a.* Add a calcium turning to 5 ml of water in a test tube. Results? Perform the burning splint test for hydrogen. Result? Add a drop of phenolphthalein indicator to the test tube contents. Result? What are the two products of this reaction? Identify the solid material formed.

b. Add several pieces of magnesium turnings (about 1 g) to 5 ml of water in a test tube. Result? Heat the test tube almost to boiling. Do you see any evidence of gas evolution? If so, what gas? Cool. Add 2 drops of phenolphthalein indicator to the contents of the test tube. Result? What probable product is indicated? What do the results of a and b indicate about the relative reactivity of magnesium and calcium?

Observations: a. The gas evolved 'barks' indicating production of H_2. Phenolphthalein turns red indicating production of $Ca(OH)_2$, the solid slightly soluble product.

b. Some slight bubbling, the gas probably being H_2. Phenolphthalein turns pink indicating production of $Mg(OH)_2$. Calcium is more active than magnesium.

2. Set up a carbon dioxide generator according to Part 2, Experiment 32. Bubble the carbon dioxide from the generator through a test tube half full of limewater. Result? Explain. Continue to bubble the gas through the contents of the test tube until a clear solution results. Explain. Heat the contents of the test tube to boiling. Result? Explain.

Observations: A white precipitate forms due to reaction between $Ca(OH)_2$ and CO_2 (or carbonic acid). This precipitate dissolves due to the formation of soluble calcium hydrogen carbonate. Boiling this solution results in the decomposition of the calcium hydrogen carbonate and the reappearance of the calcium carbonate precipitate.

3. Test 5 ml samples of the saturated solutions of the hydroxides of calcium, magnesium, and barium with Hydrion papers, both full range and the appropriate short range. What is the pH of each solution? What do these results indicate about the relative alkalinity of these compounds? Consult the Table of Solubilities in the Appendix. What trend in the solubilities of these hydroxides is indicated by the results of the pH tests?

Observations: The short range Hydrion paper pH 11-13 indicates that $Mg(OH)_2$ has a pH of approximately 11, $Ca(OH)_2$ of approximately 12 and $Ba(OH)_2$ of approximately 13, their alkalinity increasing in this order. The $Ba(OH)_2$ is most soluble, $Ca(OH)_2$ being slightly soluble, and $Mg(OH)_2$ being practically insoluble.

4. To 1 g of magnesium sulfate add 5 ml of water. Stopper and shake. Result? Repeat with separate 1 g samples of calcium sulfate and barium sulfate. Results? If complete solution does not take place, filter the test tube contents. Perform the flame tests (see Experiment 45) to determine whether some solution has taken place. Results? What trend in the solubilities of the sulfates of these metals is indicated by the results of these procedures? Confirm this by consulting the Table of Solubilities in the Appendix. Consult MODERN CHEMISTRY for the solubility products of the sulfates of calcium and barium and record the values.

Observations: $MgSO_4$ is very soluble, $CaSO_4$ is slightly soluble, and $BaSO_4$ is practically insoluble. The respective K_{sp}'s are $CaSO_4 = 6.1 \times 10^{-5}$; $BaSO_4 = 1.1 \times 10^{-10}$.

5. Add 1 ml of sodium carbonate solution to 5 ml of magnesium chloride solution. Result? Repeat with solutions of calcium chloride and barium chloride. Results? What conclusion can be reached about the solubilities of the carbonates of these metals? Check your results against the Table of Solubility Products in MODERN CHEMISTRY.

Observations: Precipitates of the carbonates result in all cases. The values in the K_{sp} Table are: $CaCO_3 = 4.8 \times 10^{-9}$ $BaCO_3 = 4.9 \times 10^{-9}$, $MgCO_3 = 2.5 \times 10^{-5}$.

FURTHER EXPERIMENTATION: Investigate the comparative solubilities of the carbonates by making the stock solutions just alkaline with ammonia-water solution, and using a solution of ammonium carbonate as the precipitating agent.

EQUATIONS: Write ionic equations where appropriate.

1. Calcium and cold water $Ca + 2H_2O \rightarrow Ca(OH)_2 + H_2(g)$
2. Magnesium and hot water $Mg + 2H_2O \rightarrow Mg(OH)_2 + H_2(g)$
3. Reactions in Part 2

$Ca^{++} + 2OH^- + CO_2 \rightarrow Ca^{++}CO_3^{--}(s) + H_2O$

$Ca^{++}CO_3^{--}(s) + H_2O + CO_2 \rightarrow Ca^{++} + 2HCO_3^-$

$Ca^{++} + 2HCO_3 \rightarrow CaCO_3(s) + H_2O + CO_2(g)$

4. Reactions in Part 5

$Mg^{++} + CO_3^{--} \rightarrow Mg^{++}CO_3^{--}(s)$

$Ca^{++} + CO_3^{--} \rightarrow Ca^{++}CO_3^{--}(s)$

$Ba^{++} + CO_3^{--} \rightarrow Ba^{++}CO_3^{--}(s)$

Experiment 47

NAME . . . . . PERIOD . . . . . DATE . . . . . .

INSTRUCTOR'S COMMENTS . . . . . .

Metallurgy of the Transition Metals

PURPOSE: To show how ores may undergo beneficiation and how metals are recovered from their ores.

APPARATUS: Balance, platform; beaker, 150 ml; burner and tubing; clamp, buret; glass bend; graduated cylinder (100 ml); magnet; ring stand; rubber stopper No. 2, one-hole; test tubes; tubing, delivery.

ADDITIONAL APPARATUS for INSTRUCTOR'S EXPERIMENT: Burner and tubing; clamp, buret; glass bends (2); glass tubing, Pyrex, large diameter (a large test tube with curved end removed will do); ring stand; stoppers, rubber, No. 4, 1-hole, No. 2, 2-hole; suction pump or aspirator; test tube; tubing delivery.

MATERIAL: Charcoal, wood, powdered; copper(II) carbonate; iron(II) (III) oxide (magnetite), powdered; lead(II) (III) oxide; mineral oil; sand; taconite (powdered); solution of calcium hydroxide.

ADDITIONAL MATERIAL for INSTRUCTOR'S EXPERIMENT: Lead(II) sulfide or zinc sulfide, powdered (ore samples will do); sulfuric acid, dilute (1:6); solution of potassium permanganate (0.2 *M*).

SUGGESTION: Samples of powdered taconite, and of powdered, crude sulfide ores may be obtained from metallurgical companies.

INTRODUCTION: Ores, particularly the low-grade ores, must be enriched or concentrated. The general name for the processes involved in beneficiation. The most common ores of the transition metals are oxides, sulfides, and carbonates. Oxides are generally reduced with carbon. Sulfides and carbonates are converted to oxides by heating them with excess air. The sulfides change to oxides, generally with the release of sulfur dioxide. The carbonates decompose to the oxides with the release of carbon dioxide.

PROCEDURE: 1. *a.* Mix 5 grams of powdered magnetite with 5 grams of sand on a piece of paper. Insert a magnet into the mixture. Result? Attempt to separate the components of samples of powdered taconite (Fe_2O_3 and Fe_3O_4) with a magnet. Result?

b. Mix 2.5 grams of powdered lead(II)(III) oxide, Pb_3O_4 with 2.5 grams of fine sand. Pour the mixture into a graduated cylinder and add 75 ml of water and 25 ml of mineral oil. Shake. Result? What name is given to this process?

Observations: a. The magnetite clings to and is removed by the magnet. The Fe_3O_4 is also removed from powdered taconite by the magnet.

b. The Pb_3O_4 separates from the sand and is suspended in the oil. This process is called flotation (or froth-flotation).

2. Set up the apparatus shown in Fig. 47-1. Place 3 grams of copper(II) carbonate in the Pyrex test tube and half fill the other test tube with limewater. Heat the copper (II) carbonate until a permanent change in color occurs. Result? What has happened to the limewater? Explain. What name is given to this process? Permit the contents of the test tube to cool.

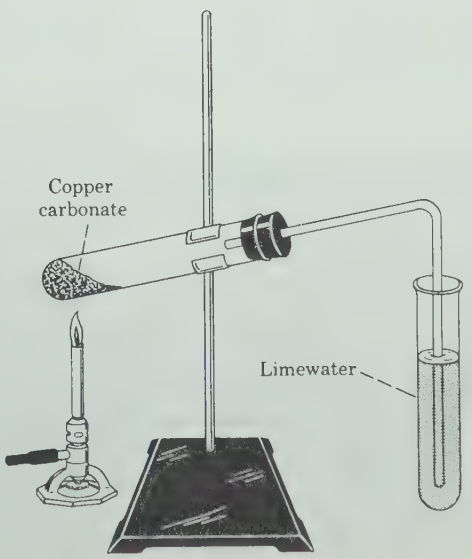

Fig. 47-1

Observations: The green $CuCO_3$ changes to black CuO. The limewater turns milky due to the evolution of CO_2. The process is called roasting (or calcining).

3. Mix the cool contents of the test tube from Part 2 with 5 grams of powdered charcoal. Refill the second test tube half-full of limewater. Set up the apparatus (Fig. 47-1) again and heat the solid mixture. Result in the limewater? Explain. Remove the delivery tube and continue to heat the solid contents of the first test tube until they have been heated for a total of 5 minutes. Permit the contents to cool and pour them into a beaker of cold water. Result? Examine the solid material left in the bottom of the beaker. Result? What process did the copper compound undergo? What process did the carbon undergo?

Observations: The limewater turns milky due to production of CO_2. The carbon floats to the top of the water and a red deposit of Cu sinks to the bottom of the beaker. The CuO underwent reduction and the C was oxidized.

4. INSTRUCTOR EXPERIMENT: Place 5 grams of either zinc sulfide or lead(II) sulfide in the central portion of the piece of Pyrex tubing, next to the retaining glass wool. Set up the remainder of the apparatus according to Fig. 47-2, half-filling the side-arm test tube with an acidified (H_2SO_4) solution of potassium permanganate. Turn on the suction or aspirator and heat the sulfide strongly. Result? What gas was produced by this process? Explain. What name

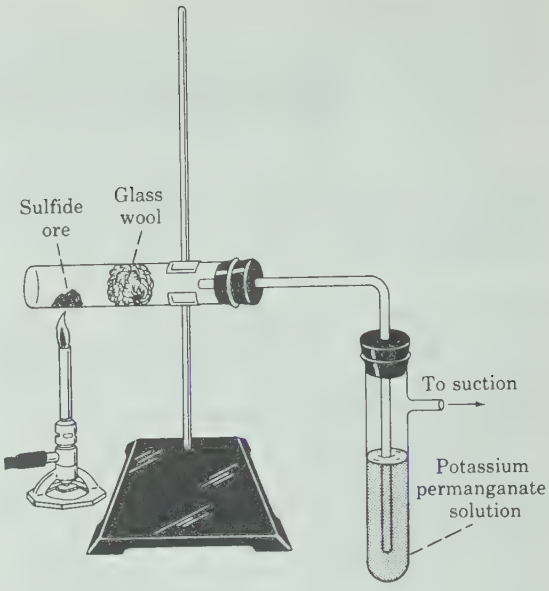

Fig. 47-2

is given to this process? What is the solid product left in the Pyrex tube? Continue the process for 5 minutes and then reduce the solid residue with carbon as in Part 3.

Observations: The $KMnO_4$ solution is bleached due to production of SO_2. The process is called roasting. The solid left is ZnO. Reduction of this ZnO with C produces Zn.

EQUATIONS

1. $CuCO_3$ (heated) → $CuO + CO_2(g)$
2. $Ca(OH)_2 + CO_2$ → $CaCO_3(s) + H_2O$
3. $2\,CuO + C$ → $2Cu + CO_2(g)$
4. $2\,ZnS + 3\,O_2$ → $2ZnO + 3SO_2(g)$
5. $ZnO + C$ → $Zn + CO(g)$

Experiment 48

NAME ..... PERIOD ..... DATE .....

INSTRUCTOR'S COMMENTS .....

Tests for Iron (II) and Iron (III) Ions

PURPOSE: To learn how to identify iron-(II) and iron(III) ions in solution.

APPARATUS: Graduated cylinder; test tubes.

MATERIAL: Solutions of: iron(III) chloride (0.1 *M*); iron(II) sulfate (0.1 *M*) or iron(II) ammonium sulfate (0.1 *M*) or iron-(II) ammonium sulfate (0.1 *M*); potassium hexacyanoferrate(III) (0.1 *M*); potassium hexacyanoferrate(II) (0.1 *M*); potassium thiocyanate (0.2 *M*).

SUGGESTION: Iron(II) ammonium sulfate is perhaps a better source of Fe^{++} ions than iron(II) sulfate. The solution of iron(II) ammonium sulfate should contain 10 ml of concentrated sulfuric acid per liter. If iron-(II) sulfate is used, 10 ml of concentrated sulfuric acid per liter of solution together with an iron nail should be added. Either solution should be freshly prepared.

INTRODUCTION: In the identification tests for the Fe^{++} and Fe^{+3} ions, we shall use the complex hexacyanoferrate(III), $Fe(CN)_6^{-3}$, and hexacyanoferrate(II), $Fe(CN)_6^{-4}$, ions. The complex ion charges clearly indicate the difference in the oxidation number of the iron present in the two complexes. The (CN) group in each complex has a charge of –1. Thus iron(II) is present in the hexacyanoferrate(II) group, $[Fe^{++}(CN^-)_6]^{-4}$. Iron(III) is present in the hexacyanoferrate(III) group, $[Fe^{-3}(CN^-)_6]^{-3}$. A deep-blue precipitate results when either complex ion combines with iron in a different oxidation state from that present in the complex. The deep-blue color of the precipitate is due to the presence of iron in *both* oxidation states. This provides us with the means of identifying either iron ion. If the deep-blue precipitate is formed on addition of the $[Fe(CN)_6]^{-4}$ complex, the iron ion responsible must be the iron(III) ion. Similarly, the deep-blue precipitate formed with the $[Fe(CN)_6]^{-3}$ complex indicates the presence of the iron-(II) ion.

Both deep-blue precipitates are now recognized as having the same composition. The potassium salts of the complex ions are commonly used, in which case the deep-blue precipitate may be considered to have the composition, $KFeFe(CN)_6 \cdot H_2O$.

The thiocyanate ion, SCN^-, provides an excellent confirming test for the Fe^{13} ion. The soluble $FeSCN^{++}$ complex is formed, imparting a rich blood-red color to the solution.

PROCEDURE: 1. To a 5-ml test solution of freshly prepared $FeSO_4$, or $Fe(NH_4)_2(SO_4)_2$, add 1 ml of $K_4Fe(CN)_6$ solution. Observe the color of the precipitate which first forms. If the $FeSO_4$ solution were thoroughly reduced, the precipitate would be white and would be considered to have the composition, $K_2FeFe(CN)_6$. Explain the lack of color. Compare the color with that which develops after the precipitate stands for a few minutes. Result? Explain. Was the initial color, of the precipitate which formed, pure white? If not, explain the presence of any coloration.

Observations: The precipitate is white. No Fe^{+++} is present. On exposure to air the precipitate starts to turn blue due to oxidation. If the initial precipitate is not pure white, some oxidation of Fe^{++} may have taken place and the Fe^{+++} ion and $Fe(CN)_6^{-4}$ give a brown solution.

2. To a second 5-ml test solution of iron(II) sulfate, add 3 or 4 drops of a solution of potassium thiocyanate, KSCN. Result? Remember that tests in chemistry which give negative results are of no value for identification.

Observations: No color or precipitate forms.

3. To a third 5-ml test solution of iron(II) sulfate, add 1 ml of $K_3Fe(CN)_6$ solution. Result? This precipitate, traditionally called Turnbull's blue, has the approximate composition, $KFeFe(CN)_6 \cdot H_2O$. What advantage does this test have over that of Part I in the identification of the Fe^{++} ion?

Observations: A deep blue precipitate forms. The white precipitate in Part I may not result if Fe^{+++} ions are present.

4. To a 5-ml test solution of iron(III) chloride, add 1 ml of the $K_3Fe(CN)_6$ solution. Result? Does a precipitate form? Do you consider this a conclusive test for the Fe^{-3} ion?

Observations: A precipitate does not result. The solution may be green and the test is inconclusive for the Fe^{+++} ion.

5. Add, to a second 5-ml test solution of iron(III) chloride, 1 ml of the $K_4Fe(CN)_6$ solution. Result? This precipitate, which is traditionally called Prussian blue, may be considered to have the same composition as that formed in Part 3, $KFeFe(CN)_6 \cdot H_2O$. Why may this test be considered conclusive in the identification of the Fe^{+3} ion?

Observations: A dark blue precipitate forms. This is conclusive test for the Fe^{+3} ion because the pigment dark blue color always develops in the presence of iron in two different states of oxidation.

6. To a third 5-ml test solution of iron(III) chloride, add 2 or 3 drops of the KSCN solution. Result? In view of the results in Part 2, do you consider this to be a conclusive test for the Fe^{+3} ion?

Observations: A blood-red solution of the $FeSCN^{++}$ ion results. This is a conclusive test for the Fe^{+3} ion because of the development of a characteristic color even in the presence of relatively small concentration of the ion. This test is often used to confirm the $K_4Fe(CN)_6$ test.

Record the results of each test in the Data Table. Give color of the precipitate or solution formed.

QUESTIONS: 1. State specifically how you would make a decisive test for an iron(III) salt. Add potassium hexacyanoferrate(II) to the solution suspected of containing the iron(III) salt. The formation of a deep blue precipitate identifies the iron(III) ion. Confirm by adding KSCN to a second test solution. Formation of a blood-red solution confirms the presence of an iron(III) salt.

2. Which test for iron(II) ions is decisive? The formation of a dark blue precipitate on addition of potassium hexacyanoferrate(III) to a solution containing iron(II) ions is the decisive test. A deep blue precipitate forms.

3. Why must an iron(II) sulfate solution be freshly prepared? The iron(II) ion rapidly oxidizes to the iron(III) ion in water solution.

4. Suppose you have a solution containing both an iron(II) salt and an iron(III) salt, how would you proceed to identify both Fe^{++} and Fe^{+3} ions in this solution? Potassium hexacyanoferrate(III) would be used to detect the iron(II) ion and potassium hexacyanoferrate(II) would be used to identify the iron(III) ion without interference.

DATA TABLE

Iron ion	Hexacyanoferrate(II) complex $Fe(CN)_6^{-4}$	Hexacyanoferrate(III) complex $Fe(CN)_6^{-3}$	Thiocyanate complex SCN^-
Fe^{++}	white ppt.	dark blue ppt.	no visible result
Fe^{+3}	dark blue ppt.	brown solution	blood-red solution

EQUATIONS

1. $Fe^{++} + Kl + Fe(CN)_6^{-3} + H_2O \rightarrow KFeFe(CN)_6 \cdot H_2O(s)$

2. $Fe^{+++} + Kl + Fe(CN)_6^{-4} + H_2O \rightarrow KFeFe(CN)_6 \cdot H_2O(s)$

3. $Fe^{+++} + SCN^- \rightarrow FeSCN^{++}$

Experiment 49

NAME. . . . . PERIOD . . . . . DATE . . . . . .

INSTRUCTOR'S COMMENTS . . . . . .

Oxidation States of Transition Elements

PURPOSE: To show that typical transition metals may exist in a variety of oxidation states, each with characteristic colors. To show how these oxidation states may be changed in oxidation-reduction reactions.

APPARATUS: Balance, platform; beaker, 150 ml; burner and tubing; clamp, test tube; dish, evaporating; funnel; graduated cylinder; ring, iron; ring stand; rubber stopper, solid No. 2; test tubes; wire gauze.

MATERIAL: Iron(II) sulfate; manganese dioxide; potassium nitrate; sodium hydroxide; zinc, mossy; filter paper; litmus paper, blue; ammonia-water solution, dilute (1:4); hydrochloric acid, dilute (1:4); nitric acid, concentrated, dilute (1:5); sulfuric acid, dilute (1:6); solutions of: chromium(III) sulfate (0.16 *M*); hydrogen peroxide (3%); iron(III) chloride (0.1 *M*); manganese sulfate (0.25 *M*); potassium chromate (0.25 *M*); potassium hexacyanoferrate(II) (0.1 *M*); potassium hexacyanoferrate(III) (0.1 *M*); potassium permanganate (0.1 *M*); potassium thiocyanate (0.2 *M*); sodium hydroxide (6 *M*); starch (colloidal); tin(II) chloride (0.1 *M*).

INTRODUCTION: The transition elements exhibit variable oxidation states and strong color. In the first-row transition metals several 4*s* and 3*d* electrons are available to be transferred to or shared with other substances. Thus several oxidation states become possible. The color of transition metals and their compounds is due to the 3*d* electrons in their structure. The compounds of chromium are all colored, the name of the element coming from the Greek word, *chroma*, meaning color. Chromium occurs in oxidation states +2, +3, and +6. Manganese compounds are also colored. Manganese occurs in oxidation states +2, +3, +4, +6, and +7. The only stable cation of manganese is the pink Mn^{++} ion. Manganates, such as K_2MnO_4, are green in color and are stable only in alkaline solutions. Permanganates, such as $KMnO_4$, are strong oxidizing agents. The permanganate ion is purple. Acid solutions of permanganate ions are useful oxidizing agents. The permanganate ion in alkaline solution may be changed to manganese dioxide, produced with a brown to black color.

The hydrated Fe^{++} ion appears to be pale green. The hydrated Fe^{+++} ion usually undergoes hydrolysis so that its solutions have a yellow-brown color. The changes in the oxidation states observed in this experiment illustrate the ease or difficulty with which the transition elements undergo changes in their oxidation states.

PROCEDURE: 1. *Chromium. a.* Add 5 ml of ammonia-water solution to 5 ml of chromium(III) sulfate solution. Result? Repeat this operation using 5 ml of sodium hydroxide solution. Result? Add an excess of sodium hydroxide solution until solution occurs. Which property of chromium(III) hydroxide is shown by this behavior? What is the oxidation state of chromium in the chromite ion (represented as either $Cr(OH)_4^-$; or the dehydrated CrO_2^-)? Save this solution for Part *c*.

Observations: Green precipitate of $Cr(OH)_3$ results. Green precipitate forms which dissolves in excess NaOH forming green chromite ions, CrO_2^-. It is amphiprotic (amphoteric). The oxidation state is +3.

b. To 5 ml of potassium chromate solution add hydrochloric acid until a definite color change occurs. Result? Explain. (Refer to the chromate-dichromate equilibrium equation in Experiment 38.) To half of this solution add ammonia-water solution until the original color is restored. Explain. What is the oxidation state of chromium in potassium chromate? What is its oxidation state in the ion produced in the acid solution?

Observations: The solution turns to the orange color of $Cr_2O_7^=$ ion. The hydronium ion concentration displaces the equilibrium toward production of $Cr_2O_7^=$ ions. The addition of OH^- removes the H_3O^+ ion displacing the equilibrium toward formation of $CrO_4^=$ ions. The oxidation state of Cr in the $Cr_2O_7^=$ ion is + 6.

c. To 5 ml of the alkaline chromite solution (from Part *a*) add hydrogen peroxide until a color change occurs. Result? To

which chromium ion has the chromite ion been changed? What did the hydrogen peroxide do to the chromite ion?

Observations: The green color of the CrO_2^- ion changes to the yellow CrO_4^{--} ion. The hydrogen peroxide oxidized the Cr changing its oxidation state from + 3 to + 6.

d. Repeat the first step in Part *b* using sulfuric acid until a definite color change occurs. Result? To this solution add hydrogen peroxide until another color change occurs. Result? To which ion of chromium was the dichromate ion changed? Was it oxidized or reduced? What change in oxidation state did the chromium undergo?

Observations: The orange color of $Cr_2O_7^{--}$ results. Excess peroxide changes the dichromate to form unstable perchromic acid which decomposes to form Cr^{+++}, H_2O, and O_2, and the solution turns green. The Cr changed from its oxidation state of + 6 and was reduced to its oxidation state of + 3 in Cr^{+++}.

e. To 5 ml of chromium(III) sulfate solution add 2 pieces of mossy zinc. Heat gently until a color change occurs. Result? This color is characteristic of the chromium(II) ion. Decant the solution and shake it with the air in the test tube until another color change occurs. Result? In which oxidation state is chromium stable in the presence of air?

Observations: The color changes to bright blue of the hydrated ion, $[Cr(H_2O)_6]^{++}$. On shaking with air, the green color of Cr^{+++} ion reappears due to oxidation of the Cr^{++} ion.

2. *Manganese. a.* Add 5 ml of sodium hydroxide solution to 5 ml of manganese sulfate solution. Result? Add 10 ml more of sodium hydroxide solution. Result? What does this indicate about the nature of manganese(II) hydroxide?

Observations: A faintly pink precipitate of $Mn(OH)_2$ is produced. Since this does not dissolve in excess NaOH, $Mn(OH)_2$ is not amphiprotic.

b. Mix 1 g of manganese dioxide with 1.5 g of sodium hydroxide and 1.5 g of potassium nitrate in an evaporating dish. Heat until the mass melts and then let it cool. Add water to the dish and stir. Filter the contents of the dish into a cylinder half-filled with water. What is the color of the filtrate? This is the characteristic color of the manganate ion, MnO_4^{--}. To 10 ml of this solution add nitric acid until the solution is acid to litmus paper. Filter. What is the color of the filtrate? This is characteristic of the permanganate ion, MnO_4^-. The brown deposit left on the filter paper is manganese dioxide. What is the oxidation state of manganese in manganese dioxide, manganate ion, and permanganate ion?

Observations: The filtrate is green. The filtrate is purple (violet). Oxidation states of manganese are: $MnO_2 = +4$; $MnO_4^- = +6$; $MnO_4^- = +7$.

c. To 5 ml of potassium iodide solution add 3 ml of sulfuric acid. Add 1 ml of potassium permanganate solution and stir. Result? Add 5 ml of collodial starch to the contents. Result? This identifies free (molecular) iodine.

Observations: The violet color of the permanganate disappears leaving a solution which is yellow with free I_2. Colloidal starch changes to blue-black.

3. *Iron. a.* Make a solution of iron(II) sulfate by dissolving 1 g or iron(II) sulfate in 30 ml of cold water. Do not agitate the solution unnecessarily, and do not apply heat. Divide the solution into three equal parts, using a Pyrex test tube for one part which is to be heated. To the portion in the Pyrex test tube, add 2 ml of dilute sulfuric acid and 1 ml of concentrated nitric acid. Boil the mixture *carefully* for 2 minutes. *Recall previous cautions in connection with heating corrosive liquids in test tubes.* When cool, test the liquid for iron(III) ions. Result? What purpose did the H_2SO_4, serve? What purpose did the HNO_3 serve? Had you used a solution of $Fe(NO_3)_2$ instead of $FeSO_4$, which acid could have been omitted? Why?

Observations: The test for Fe^{+++} is positive. The sulfuric acid was used as the acid having the corresponding sulfate ion. The HNO_3 was the oxidizing agent. If $Fe(NO_3)_2$ had been used, HNO_3 would have met both requirements for oxidation and H_2SO_4 could have been omitted

b. To the second portion of the iron(II) sulfate solution, add 2 ml of dilute sulfuric acid and about 4 ml of hydrogen peroxide solution. Shake the solution for a minute or two, and then test it for the presence of iron(III) ions. Result? Can you name

both products of this reaction? Try the equation.

Observations: The test for Fe^{+++} is positive. Iron(III) sulfate and water are the products. $2\,FeSO_4 + H_2SO_4 + H_2O_2 \rightarrow Fe_2(SO_4)_3 + 2\,H_2O$.

c. Shake the thrid portion of the iron(II) sulfate solution thoroughly for two minutes. Let it stand in the test tube rack for a few minutes. Then shake it thoroughly again. Now test it for the presence of iron(III) ions. What is the oxidizing agent in this action? Observe any change in appearance of the contents of the tube. Basic iron(III) sulfate, $Fe(OH)SO_4$, is formed and is only sparingly soluble. Set the tube aside to see if a brown precipitate finally appears. Can you write the equation for this reaction?

Observations: Continuous shaking produces the brown precipitate of basic iron(III) sulfate, $Fe(OH)SO_4(s)$, the oxygen of the air acting as oxidizing agent. Other tests may be used to show production of the Fe^{+3} ion.

$4\,FeSO_4 + O_2 + 2H_2O \rightarrow 4Fe(OH)SO_4(s)$

d. Reduction of iron(III) ions. To 5 ml of $FeCl_3$ solution, add $SnCl_2$ solution dropwise until the color of the iron(III) chloride solution has changed completely. Transfer a few drops of the solution to each of two test tubes. Test one portion for Fe^{++} ions. Result? Test the other for Fe^{+3} ions. If Fe^{+3} ions are still present, continue the reduction with the $SnCl_2$ solution and retest for Fe^{+3} ions. What is indicated by the presence of Fe^{++} ions? What ions are responsible? What change must these ions have undergone in the process? Can you write the equation?

Observations: The test with potassium hexcyanoferrate(III) ion may confirm the production of the Fe^{++} ion whose formation was indicated by the change from the yellow-brown color of Fe^{+3} to the practically colorless Fe^{++} ion. The Fe^{+3} ion has been reduced by the Fe^{+2} ion by the Sn^{++} ion which was oxidized.

$2FeCl_3 + SnCl_2 \rightarrow 2FeCl_2 + SnCl_4$

EQUATIONS: Where the unbalanced equation is given, balance by using a method for balancing oxidation-reduction equations. Complete and balance all equations.

1. $Cr^{+++} + 3\,OH^- \rightarrow Cr(OH)_3(s)$
2. $Cr(OH)_3(s) + OH^- \rightarrow CrO_2^- + 2\,H_2O$
3. $2\,CrO_4^{--} + 2\,H_3O^+ \rightarrow Cr_2O_7^{--} + 3\,H_2O$
4. $Cr_2O_7^{--} + 2\,OH^- \rightarrow 2\,CrO_4^{--} + H_2O$
5. $2\,CrO_2^- + 3\,H_2O_2 + 2\,OH^- \rightarrow 2\,CrO_4^{--} + 4\,H_2O$
6. $K_2Cr_2O_7 + 3\,H_2O_2 + 4\,H_2SO_4 \rightarrow K_2SO_4 + Cr_2(SO_4)_3 + 7\,H_2O + 3\,O_2$
7. $2\,Cr^{+++} + Zn \rightarrow 2\,Cr^{+++} + Zn^{++}$
8. $Mn^{++} + 2\,OH^- \rightarrow Mn(OH)_2(s)$
9. $MnO_2 + 2\,NaOH + KNO_3 \rightarrow Na_2MnO_4 + KNO_2 + H_2O$
10. $3\,Na_2MnO_4 + 4\,HNO_3 \rightarrow 2\,NaMnO_4 + MnO_2 + 2\,H_2O + 4\,NaNO_3$
11. $10\,KI + 8\,H_2SO_4 + 2\,KMnO_4 \rightarrow 6\,K_2SO_4 + 2\,MnSO_4 + 8\,H_2O + 5\,I_2$
12. $6\,FeSO_4 + 2\,HNO_3 + 3\,H_2SO_4 \rightarrow 2\,NO + 3\,Fe_2(SO_4)_3 + 4\,H_2O$
13. $2\,FeSO_4 + H_2O_2 + H_2SO_4 \rightarrow 2\,H_2O + Fe_2(SO_4)_3$
14. $4\,FeSO_4 + 2\,H_2O + O_2 \rightarrow 4\,Fe(OH)SO_4(s)$
15. $2\,FeCl_3 + SnCl_2 \rightarrow 2\,FeCl_2 + SnCl_4$

SUMMARY: Complete the following table, as in the example.

	Oxidation state	Electron populations		Color
		3*d*	4*s*	
Cr	0	⊘ ⊘ ⊘ ⊘ ⊘	⊘	silvery
Cr^{+++}	+ 3	⊘ ⊘ ⊘ ○ ○	○	green
CrO_2^-	+ 3	⊘ ⊘ ⊘ ○ ○	○	green
CrO_4^{--}	+ 6	○ ○ ○ ○ ○	○	yellow
$Cr_2O_7^{--}$	+ 6	○ ○ ○ ○ ○	○	orange
Mn	0	⊘ ⊘ ⊘ ⊘ ⊘	⊗	silvery
Mn^{++}	+ 2	⊘ ⊘ ⊘ ⊘ ⊘	○	pink
MnO_2	+ 4	⊘ ⊘ ⊘ ○ ○	○	black
MnO_4^{--}	+ 6	⊘ ○ ○ ○ ○	○	green
MnO_4^-	+ 7	○ ○ ○ ○ ○	○	purple
Fe	0	⊗ ⊘ ⊘ ⊘ ⊘	⊗	silvery
Fe^{++}	+ 2	⊗ ⊘ ⊘ ⊘ ⊘	○	pale green (colorless)
Fe^{+++}	+ 3	⊘ ⊘ ⊘ ⊘ ⊘	○	yellow-brown

Experiment 50

NAME . . . . . PERIOD . . . . . DATE . . . . . .

INSTRUCTOR'S COMMENTS . . . . . .

Complex Ions of Copper and Silver

PURPOSE: To study some complex ions of copper and silver and to show how certain combinations are used in chemical analysis.

APPARATUS: Graduated cylinder; medicine dropper; test tubes.

MATERIAL: Acetic acid, glacial; litmus paper, red; ammonia-water solution, concentrated; solutions of: copper(II) sulfate (0.1 *M*); potassium bromide (0.2 *M*); potassium cyanide (0.2 *M*); potassium hexacyanoferrate(II) (0.1 *M*); potassium iodide (0.2 *M*); silver nitrate (0.2 *M*); sodium chloride (0.2 *M*); sodium hydroxide (1 *M*); sodium thiosulfate (0.2 *M*).

ADDITIONAL MATERIAL for INSTRUCTOR'S EXPERIMENT: Solution of hydrogen sulfide (saturated).

CAUTION: *Potassium cyanide is a deadly poison. Do not get it on your hands. It should be used only under the direct supervision of the instructor.*

INTRODUCTION: Transition metals form complex ions with ammonia molecules (NH_3), cyanide ions (CN^-), thiosulfate ions ($S_2O_3^{--}$), and thiocyanate ions (SCN^-). The transition metal is the central atom in each case. Refer to Experiment 40 which deals with complex ion equilibria and the small values of their ionization constants. In this experiment, further use of complex ions for identification and analysis will be explored. You will also investigate the role of complex silver ions in the identification of silver halogenides, several of which are used in photography. Use the following table of solubility products and ionization constants for reference.

PROCEDURE: 1. *Copper. a.* To 5 ml of copper(II) sulfate solution add 2 or 3 ml of sodium hydroxide solution. What is the precipitate which forms? Describe its color and appearance. Add NaOH dropwise until the solution is alkaline (litmus test). Is any change observed? Write the equation.

Observations: Pale blue precipitate of $Cu(OH)_2$ forms. Ir gives no evidence of solution in excess NaOH.

$CuSo_4 + 2\ NaOH \rightarrow Na_2SO_4 + Cu(OH)_2$

b. To 5 ml of copper(II) sulfate solution, add 2 or 3 drops of ammonia-water solution. Result? Name the precipitate. Now add ammonia-water in excess. (How can you determine this?) What two changes do you observe? Recall the results in Part *a*. Can these changes be attributed to an excess of OH^- ions? The intense color is characteristic of what complex ion? What precaution should you observe in using this as a qualitative test for copper(II) ion?

Observations: The pale blue precipitate of copper(II) hydroxide forms first. Ammonia water added in excess and detected by use of litmus paper causes the precipitate to dissolve to form a dark blue solution. This change is not due to excess OH^- ion (amphiprotism.) The color is characteristic of the complex $[Cu(NH_3)_4]^{++}$ ion. In using this test, it is necessary to make sure that excess ammonia-water has been added.

c. Acidify 5 ml of copper(II) sulfate solution by adding 3 drops of glacial acetic acid. Then add 5 drops of a solution of $K_4Fe(CN)_6$. Result? Describe the precipitate. It may be considered to be $Cu_2Fe(CN)_6$. Recall the test for the Fe^{+++} ion. Is this a reliable, qualitative test for copper(II) ions which may be used to detect traces of copper?

Precipitate or complex ion	K_{sp}	K_i
½ Ag_2O ($Ag^+ + OH^-$)	2×10^{-8}	
AgCl	1.2×10^{-10}	
AgBr	4.8×10^{-13}	
AgI	1.5×10^{-16}	
$Ag(NH_3)_2^+$		6.8×10^{-8}
$Ag(S_2O_3)_2^{-3}$		6×10^{-14}
$Ag(CN)_2$		2×10^{-19}
CuS	8.5×10^{-45}	
$Cu(OH)_2$	5.6×10^{-20}	
$Cu(NH_3)_4^{++}$		5×10^{-14}
$Cu(CN)_4^{-3}$		2×10^{-27}

Observations: The precipitate is red. This is a reliable test for Cu(II) ions.

d. INSTRUCTOR'S EXPERIMENT. To 5 ml of copper(II) sulfate solution, add 5

ml of saturated hydrogen sulfide solution. Result? Describe the precipitate. Under the hood, add potassium cyanide solution dropwise to 5 ml of copper(II) sulfate solution until no further precipitation is evident.

CAUTION: *Cyanogen gas, $(CN)_2$ is formed and is extremely poisonous.* Describe the action and write the equation. Continue the addition until an excess of KCN solution is present. Result? What complex ion is formed? Write the equation for the reaction. Divide the solution into two portions. To one, add H_2S solution. Result? Recall result of use of H_2S only. To the other portion, add ammonia-water solution in excess. Result? Recall the result in Part *b*. Account for any differences observed.

Observations: Black CuS is precipitated. The precipitate first formed dissolves in excess KCN and eventually the pale blue $Cu(CN)_2^-$ ion is formed.

$$2\,Cu^{++} + 4\,CN^- \rightarrow 2\,CuCN(s) + (CN)_2(g)$$

$$CuCN + CN^- \rightarrow [Cu(CN)_2]^-$$

or $2\,Cu^{++} + 6\,CN^- \rightarrow 2[Cu(CN)_2]^- + C_2N_2$

No precipitate results when H_2S is added to the $[Cu(CN)_2]^-$ ion. Ammonia-water does not produce the deep-blue $[Cu(NH_3)_4]^{++}$ because the $[Cu(CN)_2]^-$ is a very stable complex and the Cu has an oxidation state of +1.

2. *Silver. a.* To separate 5 ml portions of silver nitrate solution, add an equal volume of the solution of the following reagents: (1) sodium chloride; (2) potassium bromide; (3) potassium iodide. Note the colors of the precipitates formed. Name the precipitates. Save these for Parts *b*, *c*, and *d*.

Observations: Silver nitrate: white; silver bromide: pale yellow; silver iodide: yellow.

b. Divide the precipitate in (*a*, 1) into three equal portions. To the first portion add 5 ml of ammonia-water solution. To the second portion add 5 ml of sodium thiosulfate solution. To the third portion add 5 ml of potassium cyanide solution. Stir the test tube contents in each case. Results? Identify the complex ions produced.

Observations: Silver chloride dissolves in all three; silver bromide dissolves slightly in ammonia-water and completely in the other two; silver iodide dissolves only in KCN. The complex ions are $[Ag(NH_3)_2]^+$; $[Ag(CN)_2]^-$.

c. Repeat Part *b* with the precipitate from (*a*, 2). Results? Which reagents produce solutions of complex ions? Explain.

Observations: Silver bromide dissolves slightly in ammonia-water and completely in thiosulfate and potassium cyanide solutions, forming complexes which are much more stable with smaller K_i's

d. Repeat Part *b* with the precipitate from (*a*, 3). Results? Which reagents do not dissolve the precipitate? Explain. Devise a scheme for identifying the silver halogenides and briefly describe it.

Observations: Ammonia-water and the thiosulfate solutions do not dissolve AgI because it is the least soluble and has the smallest K_{sp} of the three silver halogenides. Precipitate the halide, note its color and then use the three complexing solutions to test for relative solubility. If the silver halide dissolves only in KCN, it identifies iodide; if it dissolves in both KCN and $Na_2S_2O_3$ but does not dissolve completely in ammonia-water, the halogenide ion is bromide; If the precipitate dissolves completely in ammonia-water, the halogenide is a chloride.

EQUATIONS

1. $Cu^{++} + SO_4^{--} + 2\,Na^+ + 2\,OH^- \rightarrow 2Na^+ + SO_4^{--} + Cu(OH)_2(s)$
2. $Cu^{++} + 4\,NH_3 \rightarrow [Cu(NH_3)_4]^{++}$
3. $2\,Cu^{++} + Fe(CN)_6^{-4} \rightarrow Cu_2Fe(CN)_6(s)$
4. $Cu^{++} + SO_4^{--} + 2\,H_3O^+ + S^{--} \rightarrow 2H_3O^+ + SO_4^{--} + CuS(s)$
5. $2\,Cu^{++}$ (excess) $+ 4\,CN^- \rightarrow 2CuCN(s) + (CN)_2(g)$
6. $CuCN + CN^- \rightarrow [Cu(CN)_2]^-$
7. $AgCl(s) + NH_3$ (excess) $\rightarrow [Ag(NH_3)_2]^+ + Cl^-$
8. $AgBr(s) + 2\,S_2O_3^{--} \rightarrow [Ag(S_2O_3)_2]_2^{-3} + Br^-$
9. $AgI(s) + 2\,CN^- \rightarrow [Ag(CN)_2]^- + I^-$

NAME ..... PERIOD ..... DATE .....
INSTRUCTOR'S COMMENTS .....

Experiment 51 Zinc and its Compounds

PURPOSE: To study the properties of zinc and some of its compounds.

APPARATUS: Burner and tubing; clamp, test tube; file, triangular; forceps; graduated cylinder; medicine dropper; ring, iron; ring stand; test tubes.

ADDITIONAL APPARATUS FOR INSTRUCTOR'S EXPERIMENT: Burner, Meker or Fisher, and tubing; crucible, sand.

MATERIAL: Zinc, mossy; zinc, sheet, strips, 1 cm × 5 cm; phenolphthalein indicator; sandpaper; wooden splints; ammonia-water solution, dilute (1:4); hydrochloric acid, dilute (1:4); solutions of: ammonium sulfide (0.2 *M*); hydrogen sulfide (saturated); sodium hydroxide (3 *M*); zinc sulfate (0.2 *M*).

ADDITIONAL MATERIALS FOR INSTRUCTOR'S EXPERIMENT: Ammonium chloride; iron nail; sawdust; sulfuric acid, dilute (1:6).

INTRODUCTION: Zinc is a self-protective metal and is used as a protective coating for sheet iron. Zinc reacts with acids. It also reacts with active metal hydroxides forming zincates and liberating hydrogen. Its characteristic oxidation state is + 2.

The hydroxides of some metals may act either as a base or as an acid. Zinc hydroxide is an example. When a strong acid such as hydrochloric acid is present, zinc hydroxide forms hydroxide ions. In the presence of a strong hydroxide, such as sodium hydroxide, zinc hydroxide forms zincate, ZnO_2^{--}, ions. Thus we may consider zinc hydroxide as either $Zn(OH)_2$ or zincic acid, H_2ZnO_2. The metallic hydroxides that behave in this manner are said to be amphiprotic.

PROCEDURE: 1. Sandpaper a piece of zinc and observe its color and luster. Bend it to note its flexibility. Test its hardness by attempting to scratch it with one of the sharp edges at the end of the file. Using forceps, hold a small piece of mossy zinc in the outer edge of the burner flame. Note the color it imparts to the flame as it oxidizes.

Observations: Student answer .....

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2. To 5 ml of hydrochloric acid, add 2 pieces of mossy zinc. Results? Perform the flaming splint test for hydrogen. Result? Repeat using 5 ml of sodium hydroxide solution. If there is no immediate sign of reaction, heat gently. Perform the flaming splint test for hydrogen after the reaction has been permitted to take place for at least one minute. Result?

Observations: H_2 which evolves is identified by the "bark" test. Heating the Zn + NaOH again produces H_2 identified by the "bark" test. .....

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3. To a half test tube of zinc sulfate solution, add 2 or 3 drops of sodium hydroxide solution. Result? Explain. Shake the tube and divide the contents into two parts. To the first half, add 6 or 8 drops of hydrochloric acid. Result? Explain.

To the second portion, add sodium hydroxide in slight excess. Result? What does this indicate about the nature of the hydroxide of zinc? The clear solution now contains sodium zincate, Na_2ZnO_2. Add a drop of phenolphthalein to the test tube. Then add dilute hydrochloric acid drop by drop until the excess sodium hydroxide is just neutralized. Result? Explain. What is the effect of adding an excess of hydrochloric acid?

Observations: The white precipitate of $Zn(OH)_2$ is produced. This dissolves in HCl since it acts as a metallic hydroxide. Excess NaOH dissolves the second portion, indicating the $Zn(OH)_2$ is amphiprotic (amphoteric). The phenolphthalein turns colorless when neutralization of excess NaOH has taken place. Successive further additions of HCl may first cause the $Zn(OH)_2$ precipitate to reprecipitate and then subsequently dissolve in the excess acid. .....

4. To 5 ml of zinc sulfate solution, add two or three drops of dilute hydrochloric acid and 3 ml of hydrogen sulfide solution. Result? Does zinc sulfide precipitate in an acid solution?

To 5 ml of zinc sulfate solution, add 2 ml of ammonium sulfide solution. Result? To 5 ml of zinc sulfate solution, add just enough sodium hydroxide dropwise to form a precipitate. What is this precipitate? Shake, allow the precipitate to settle, and pour off the supernatant liquid. Add dropwise to the precipitate enough ammonia-water solution to dissolve it. The very slightly ionized complex $Zn(NH_3)_4^{++}$ ion is formed, thus reducing the concentration of Zn^{++} ions so that the excess $Zn(OH)_2$ dissolves. Now add dropwise hydrogen sulfide solution in slight excess. How can you determine this? Result?

Observations: ZnS does not precipitate in acid solution. It does precipitate upon the addition of $(NH_4)_2S$ to the $ZnSO_4$ solution. The precipitate formed with NaOH is $Zn(OH)_2$. H_2S addition to $Zn(NH_3)_4^{++}$ solution results in the precipitation of ZnS. The presence of excess H_2S may be determined by the use of either phenolphthalein or litmus indicator.

5. INSTRUCTOR'S EXPERIMENT. Melt several pieces of mossy zinc in a large sand crucible. Use a Meker or Fisher burner, if available. Cover the surface of the molten metal with some powdered ammonium chloride and sawdust. Dip into the molten zinc an iron nail that has been cleaned by immersing it in dilute sulfuric acid and then letting it drain until it is dry. Why are dipping vats for galvanizing covered with sawdust and ammonium chloride? The crucible and zinc may be set aside to use with other classes or from term to term.

Observations: The sawdust and ammonium chloride are used to prevent the zinc from catching fire at the upper surface.

QUESTIONS: 1. Does metallic zinc burn in air? Zinc burns but not very readily. (Zinc dust, dropped through the burner flame, burns brilliantly).

2. What are the conditions under which ZnS may be precipitated from a soluble zinc salt? ZnS precipitates from an ammonical solution using hydrogen sulfide in slight excess. It does not precipitate in acid solution (it can be precipitated slowly but completely by H_2S in a buffered solution in which the hydronium ion concentration is $10^{-2}M$). It is also precipitated by the reagent $(NH_4)_2S$.

3. What is the effect of hydrochloric acid on a precipitate of zinc hydroxide? The zinc hydroxide dissolves in hydrochloric acid.

4. What is the effect of sodium hydroxide on a precipitate of zinc hydroxide? The zinc hydroxide precipitate dissolves in sodium hydroxide.

EQUATIONS

1. $Zn + 2NaOH \rightarrow Na_2ZnO_2 + H_2(g)$
2. $ZnSO_4 + 2NaOH \rightarrow Na_2SO_4 + Zn(OH)_2(s)$
3. $Zn(OH)_2 + HCl \rightarrow ZnCl_2 + 2H_2O$
4. $H_2ZnO_2 + 2NaOH \rightarrow Na_2ZnO_2 + 2H_2O$
5. $ZnSO_4 + (NH_4)_2S \rightarrow ZnS(s) + (NH_4)_2SO_4$
6. $Zn(OH)_2 + 4NH_3 \rightarrow Zn(NH_3)_4^{++} + 2OH^-$
7. $Zn(OH)_2 + H_2S \rightarrow ZnS(s) + 2H_2O$

Experiment 52

NAME . . . . . PERIOD . . . . . DATE . . . . . .

INSTRUCTOR'S COMMENTS . . . . . .

Cobalt Nitrate Tests

PURPOSE: To learn how to identify zinc, aluminum, and magnesium in compounds by means of the cobalt nitrate test.

APPARATUS: Beaker, 50 ml; blowpipe and tip; burner and tubing; clamp, buret; forceps; medicine dropper; ring stand.

MATERIAL: Aluminum sulfate; magnesium sulfate; zinc sulfate; charcoal block, blowpipe quality (or plaster block); unknowns containing Al, Mg, or Zn; solution of cobalt(II) nitrate (0.1 *M*).

SUGGESTION: A plaster block may be used in place of the charcoal block. To make plaster blocks, mix plaster of Paris and water to the consistency of a moderately stiff paste. Then pour enough into a flat enameled iron pan to make a layer about 1 cm deep. As soon as it begins to set or harden, cut it into blocks about 4 cm × 8 cm. Plaster blocks should be made the day before the experiment is to be performed.

INTRODUCTION: Cobalt(II) nitrate, when heated strongly, decomposes to the oxide and combines with the oxides of certain metals to form distinctly colored complexes. With zinc, for example, it forms a *zincate*; with aluminum, an *aluminate*. The use of cobalt(II) oxide as a pigment in decorating china is an example of this action. The cobalt units with the aluminum in the china to form a blue pigment when the china is fired.

The color of the magnesium residue is faint. It should be examined near a window in a good light after your eyes have been rested for a few minutes. The eyes tire of certain colors, due to retinal fatigue, and the person may fail to see the faint tint under such a condition.

Blowpipe technique: If this is your first experience in using the blowpipe, it is suggested that you practice to develop the proper technique before beginning the experiment. Close the air ports of your burner and reduce the luminous flame to a height of 3 or 4 cm. If a support is not used, hold the end of the blowpipe in the middle of the flame so the jet flame will emerge from the side slanting downward at a slight angle. Blow with a steady pressure to produce a small, pointed, sharply defined, blue flame. Clamp the charcoal or plaster block on the ring stand, or hold it with the free hand, in a plane perpendicular to the blowpipe flame. Place it so that the sharp tip of the flame just enters the depression in the block. See diagram, Fig. F-14. Keep the cheeks puffed while blowing and learn to breathe through the nostrils without interrupting the steady pressure on the blowpipe.

PROCEDURE: 1. Make a shallow hole near one end of the charcoal or plaster block with the blunt end of the forceps. Fill the depression with zinc sulfate, add a drop of water to moisten it so it will not blow away easily, and then heat it strongly with the oxidizing flame of the blowpipe. Predict the composition of the residue. What is its color when hot? After it has cooled? Add one drop of a solution of cobalt(II) nitrate to the residue and again heat the residue strongly in the oxidizing flame of the blowpipe. If the residue is not heated strongly, it may appear blue due to the dehydration of the cobalt(II) nitrate. Blue is not the correct color and indicates that the cobalt-zinc complex has not been formed. Heat strongly again. Examine the residue. Enter the colors, before and after the addition of $Co(NO_3)_2$ solution, in the Data Table.

Observations: The ZnO is canary yellow when hot and white when cold. The residue is dark green.

2. Make a second hole in the block as before and repeat the procedure, using aluminum sulfate instead of zinc sulfate. The color you observe is characteristic of aluminum and its components. Record.

Observations: The aluminum residue for the cobalt nitrate test is blue.

3. Make a third hole in the block and repeat the procedure, using magnesium sulfate. Rest your eyes, and then take the residue to the window for examination. Record.

Observations: The Mg residue is a delicate pink (flesh color).

4. Get a sample of an unknown salt from your instructor and test it as before. Record your findings in the Data Table. If unknowns are numbered, be sure to include the number of the one tested in your report in the Data Table. The unknown tested was determined to be a compound containing

Student answer

QUESTIONS: 1. Compose a statement which describes the cobalt nitrate tests as you have used them. The cobalt nitrate tests are tests for certain metals and depend on the characteristic colors produced when the oxides of these metals are heated with $Co(NO_3)_2$ in the oxidizing flame of the blowpipe. By these tests we are able to identify the following: Al – blue, Mg – flesh (pink), and Zn – green.

2. If you were given a sample of an unknown metal and were required to determine whether it is aluminum, magnesium, or zinc, what two possible methods could you describe for preparing the substance for a cobalt nitrate test? (a) Attempt to convert a portion of the sample to its oxide by heating it directly with the oxidizing flame of the blowpipe. If the oxide is formed proceed directly with the cobalt nitrate tests.

(b) React a portion of the sample with hydrochloric acid; evaporate to dryness; and proceed with the cobalt nitrate test, using the salt of the metal recovered.

DATA TABLE

Compound used	Color of residue after first heating	Color of residue after second heating
Zinc	yellow to white	dark green
Aluminum	white	blue
Magnesium	white	pink
Unknown		

Experiment 53

NAME . . . . . PERIOD . . . . . DATE . . . . . .
INSTRUCTOR'S COMMENTS . . . . . .

Separation of Lead(II), Silver, and Mercury(I) Ions

PURPOSE: To separate a mixture of the salts of lead(II), silver, and mercury(I) and identify each metallic ion.

APPARATUS: Beaker, 150 ml; burner and tubing; clamp, test tube; funnel; glass plate; graduated cylinder; ring, iron; ring stand; test tubes; wash bottle.

MATERIAL: Litmus paper, blue; filter paper; ammonia-water solution, dilute (1:4), hydrochloric acid, dilute (1:4); nitric acid, dilute (1:5); solutions of: lead(II) nitrate (0.1 *M*), mercury(I) nitrate (0.1 *M*), potassium chromate (0.1 *M*), silver nitrate (0.2 *M*), unknowns.

INTRODUCTION: In analytical chemistry, one may be required to identify a substance, or to find out what a mixture contains. In qualitative analysis, we determine *what* is present. In quantitative analysis, we find *how much* of a substance is present.

In this experiment, we shall separate a mixture of lead(II), silver, and mercury(I) compounds by means of differences in their solubilities. We make use of the fact that the chlorides of these three metals are insoluble under similar circumstances. They may be co-precipitated as chlorides. Consequently, the group of metals is known as the *insoluble chloride group*. This experiment is an introduction to the system of analysis used by chemists.

PROCEDURE: 1. *Identification of* Pb^{++}. To 5 ml of lead(II) nitrate solution, add just enough dilute hydrochloric acid to precipitate all of the lead as lead(II) chloride. This can be accomplished by first adding about 1 ml of hydrochloric acid, shaking, letting the precipitate settle, and then adding acid dropwise. The liquid at the top will be clear enough so you can tell, as the drop strikes the surface, whether any precipitate is being formed. Shake vigorously and let the precipitate settle. Pour off the supernatant liquid.

Add enough cold water to nearly fill the test tube, and shake to wash the precipitate free from any excess acid. After the bulk of the precipitate has settled, again pour off the supernatant liquid. Test the solubility of lead(II) chloride in *hot water* by adding 10 ml of hot (not boiling) water to the precipitate and shaking. To ascertain whether part, at least, of the lead chloride has dissolved, filter the solution if it is not clear. Add about 1 ml of a solution of potassium chromate to the filtrate. Result? What insoluble product is formed? What distinguishing characteristic does it have?

Observations: A yellow precipitate of $PbCrO_4$ forms.

2. *Identification of* Ag^+. To 5 ml of silver nitrate solution, add about 1 ml of dilute hydrochloric acid. Shake vigorously to coagulate the precipitate and allow to settle Add additional hydrochloric acid dropwise as necessary to insure complete precipitation of the silver chloride. When the precipitate has settled sufficiently, pour off the supernatant liquid to save time in filtering. Filter the precipitate, washing the portion that clings to the walls of the test tube into the filter cone with small additions of water from the wash bottle. Wash the precipitate on the filter paper several times with small additions of water from the wash bottle. Discard these washings (filtrates). Divide the precipitate into two parts. This may be done by puncturing the filter cone at the point and carefully washing the precipitate from one half of the filter cone (wash bottle) into a clean test tube. Wash the second half of the filter cone into a second clean test tube. Discard the wash water by decantation after the precipitates have settled. Test the solubility of one portion of the silver chloride in hot (not boiling) water. Result? Test the solubility of the other portion of silver chloride in dilute ammonia-water solution. Is there visible evidence that the precipitate is soluble in this reagent? Shake. Filter if necessary. Verify the solubility of silver chloride in the ammonia-water solution by adding dilute nitric acid dropwise to clear liquid (shake) to neutralize the ammonia. When the odor of ammonia is no longer present, touch a drop of the solution to a litmus test strip to confirm that the environment is acidic. Result? On addition of ammonia-

water solution to the silver chloride precipitate, the soluble complex, $Ag(NH_3)_2^+$, ion is formed. Interpret this procedure and the observed results in terms of this fact.

Observations: AgCl is not soluble in hot water. It does dissolve in ammonia-water solution, much of it dissolving without shaking, the rest dissolving with suitable excess of ammonia. Acidic solution turns litmus to red. Dilute nitric acid results in reprecipitation of AgCl. The ammonia-water formed the $[Ag(NH_3)_2]^+$ ion dissolving the AgCl. Addition of the HNO_3 reacts with the NH_3 and the complex ion changes back to free Ag^+ which reacts with Cl^- to reform the AgCl precipitate.

3. *Identification of* Hg_2^{++}. To 5 ml of mercury(I) nitrate solution, add enough dilute hydrochloric acid to precipitate completely the mercury as mercury(I) chloride. Filter and wash the precipitate repeatedly with cold water (wash bottle). Divide the precipitate into two parts, using the technique outlined in Part 2. Discard the wash water from each part by decantation after the precipitates have settled. Best the solubility of one part in hot (not boiling) water. Result? To the other part add 5 ml dilute ammonia-water solution. Result? Ammonia reacts with mercury(I) chloride to form insoluble *white* mercury(II) aminochloride, $HgNH_2Cl$, and metallic mercury as a *black* dispersion. This is an example of an *auto-oxidation-reduction* reaction. Carefully describe the appearance of the residue.

Observations: Hg_2Cl_2 is not soluble in hot water. Addition of ammonia-water produces a precipitate which may be either gray or black.

4. The separation of Pb^{++}, Ag^+, and Hg_2^{++}. Prepare a mixture containing solutions of lead(II), silver, and mercury(I) nitrate, about 3 ml of each. Precipitate the chlorides with dilute hydrochloric acid. Filter and wash the precipitates on the filter paper with several additions of cold water from the wash bottle. On the basis of previous tests, determine whether this filtrate should be saved or discarded. Wash the precipitate with about 10 ml of hot (not boiling) water, collecting the filtrate in a clean test tube. Pour the filtrate through the filter cone a second time. Then test the filtrate for the presence of lead. Recall the identifying test for the Pb^{++} ion in Part I. Result?

To the precipitate left on the filter, add 5 ml of dilute ammonia-water solution, collecting the filtrate. Pour the filtrate back through the filter cone a second time. Why? Test the filtrate for silver by adding dilute nitric acid until the filtrate is acidic as shown by the litmus test. Result?

Examine the residue remaining on the filter paper. Recall the action of the ammonia-water solution on mercury(I) chloride in Part 3. Result?

Observations: Addition of chromate ion to the dissolved $PbCl_2$ produces the yellow $PbCrO_4$ precipitate. Dilute HNO_3 reprecipitates the white AgCl. The residue on the filter paper is blackish-gray.

5. *The identification of an unknown.* Get a sample of an unknown solution from your instructor. Analyze the solution for the possible presence of Pb^{++}, Ag^+, and Hg_2^{++}, following the method outlined in Part 4. Report the metallic ions present. If the unknowns are numbered, be sure to include the number of the unknown tested in your report.

Unknown Report: Unknown number ....

Metallic ions present: .....

QUESTIONS: 1. How can you separate a mixture of lead(II) chloride and silver chloride? To separate a mixture of lead and silver chloride, wash the precipitate with hot water and filter, AgCl remains as the residue, $PbCl_2$ is soluble in hot water and is separated as the filtrate.

2. How can you distinguish mercury(I) chloride from silver chloride? Silver chloride is soluble in ammonia water and is removed by filtration. Mercury(I) chloride turns dark gray to black in ammonia water but remains insoluble.

3. Compose a statement which describes the *insoluble chloride group* tests as you have used them. Tests for the Ag, Pb, and Hg_2 ions depend on the insolubility of their chloride, the subsequent differences in solubility in hot water and ammonia-water solution, and the reformation of characteristic precipitates following their separation.

EQUATIONS

1. $Pb^{++} + 2Cl^- \rightarrow$ $PbCl_2(s)$
2. $Pb^{++} + CrO_4^{--} \rightarrow$ $PbCrO_4(s)$
3. $Ag^+ + Cl^- \rightarrow$ $AgCl(s)$
4. $AgCl(s) + 2NH_3 \rightarrow$ $Ag(NH_3)_2^+ + Cl^-$
5. $Ag(NH_3)_2^+ + Cl^- + 2H_3O^+ \rightarrow$ $AgCl(s) + 2NH_4^+ + 2H_2O$
6. $Hg_2^{++} + 2Cl^- \rightarrow$ $Hg_2Cl_2(s)$
7. $Hg_2Cl_2(s) + NH_3 \rightarrow$ $HgNH_2Cl(s) + Hg(s) + NH_4Cl$

Experiment 54

NAME ..... PERIOD ..... DATE .....

INSTRUCTOR'S COMMENTS .....

Aluminum and its Compounds

PURPOSE: To study aluminum and some of its compounds.

APPARATUS: Balance, platform; burner and tubing; clamp, test tube; graduated cylinder; test tubes.

ADDITIONAL APPARATUS FOR INSTRUCTOR'S EXPERIMENT: Asbestos square, with hole in center; 2 clamps, buret; 2 ring stands; sand bath, large, shallow.

MATERIAL: Aluminum, chips or granules; aluminum, foil; cotton, absorbent; litmus papers, red and blue; ammonia-water solution, dilute (1:4); hydrochloric acid, concentrated; hydrochloric acid, dilute (1:4); nitric acid, concentrated; sulfuric acid, concentrated; solutions of: aluminum chloride (0.2 *M*); aluminum sulfate (0.1 *M*); ammonium sulfide (0.3 *M*); mercury(I) nitrate (0.1 *M*); sodium carbonate (0.3 *M*); sodium hydroxide (2.5 *M*).

ADDITIONAL MATERIAL FOR INSTRUCTOR'S EXPERIMENT: Ignition mixture; magnesium ribbon; sand; thermite; asbestos paper; filter paper.

INTRODUCTION: Aluminum is a self-protective metal which rapidly becomes covered with an adherent impervious layer of aluminum oxide. Hydrochloric acid and sulfuric acid react with aluminum readily, but nitric acid does not. A strong hydroxide, such as sodium hydroxide, also reacts with aluminum, setting free hydrogen and forming sodium aluminate, $NaAlO_2$.

Aqueous solutions of most aluminum salts are acid because of the hydrolysis of the hydrated aluminum ion, $Al(H_2O)_6^{+++}$, which may be regarded as a Brønsted acid according to the following reaction:

$$Al(H_2O)_6^{+++} + H_2O \rightleftarrows Al(H_2O)_5(OH)^{++} + H_3O^+$$

Aluminum hydroxide is amphiprotic.

PROCEDURE: **1.** Rub a small piece of aluminum foil with a piece of cotton moistened in mercury(I) nitrate to amalgamate the aluminum. Results? Test the unamalgamated side of the aluminum foil for any evidence of the release of heat by the oxidation reaction. Results? What is the white, flaky material which is produced?

Observations: A powdery white deposit forms on the amalgamated surface and the unamalgamated side feels warm. The flaky white material is Al_2O_3.

2. Test 5 ml of solutions of aluminum chloride and of aluminum sulfate with red and blue litmus papers. Result? Explain.

Observations: Both salt solutions turn blue litmus to red because of the hydrolysis of the $Al(H_2O)_6^{+3}$ ion.

3. Cover 1 g of aluminum chips or granules with water in a test tube and add a few drops of concentrated hydrochloric acid. Warm the test tube, if necessary, to start the reaction. Result? What products are formed? Repeat with sulfuric and nitric acids. Compare the results.

Observations: H_2 gas is produced by reaction of the Al with HCl and H_2SO_4. There is no evidence of any reaction with HNO_3.

4. Add 1 g of aluminum chips to a test tube and then cover the metal with sodium hydroxide solution. Warm the test tube, if necessary, to start the reaction. Result? What products are formed?

Observations: H_2 gas is produced along with sodium aluminate, $NaAlO_2$.

5. To 5 ml of aluminum chloride solution, add 1 ml of ammonia-water solution. Result? What products are formed? Shake the test tube to flocculate the precipitate. Allow the precipitate to settle, and pour off the supernatant liquid. Transfer one-half of the precipitate to a second test tube. To the precipitate in one test tube, add 1 ml of sodium hydroxide solution. To the precipitate in the other test tube, add 1 ml of dilute hydrochloric acid. Compare the results. What property of aluminum hydroxide is thus illustrated?

Observations: The white gelatinous precipitate of $Al(OH)_3$ is produced, the other product is ammonium chloride. The portions of $Al(OH)_3$ dissolve respectively in HCl and NaOH. These reactions show that $Al(OH)_3$ is amphiprotic.

6. Add 1 ml of sodium carbonate solution to 5 ml of aluminum sulfate solution. You might expect aluminum carbonate to be formed in this case, but the precipitate that forms is aluminum hydroxide. Why? (Remember that aluminum hydroxide is a weak hydroxide and carbonic acid is a weak acid.) Explain the reaction.

Observations: If aluminum carbonate were formed initially it is immediately hydrolyzed causing $Al(OH)_3$ to form.

7. Add 5 ml of ammonium sulfide solution to 5 ml of aluminum sulfate solution. Re-

sult? Is the insoluble product the same as that formed in Part 6? Why isn't aluminum sulfide produced?

Observations: The white precipitate is $Al(OH)_3$, the same as in Part 6. Al_2S_3 is not formed from solution since it is a salt of a weak acid and a weak hydroxide therefore undergoing hydrolysis.

8. INSTRUCTOR'S EXPERIMENT. Set up the apparatus as shown in Fig. 54-1. Spread a layer of dry sand over asbestos paper on the table to protect the table top. In addition, nearly fill a large, shallow sand bath with dry sand. Fold a piece of filter paper into a cone and insert it in the hole in the asbestos board. Nearly fill the cone with thermite, and add starting mixture (powdered magnesium, 1 part, to barium peroxide, 9 parts), and magnesium ribbon as shown. When all is ready, have the class get at least 3 meters away from the apparatus.

Caution: *Be very careful in executing this experiment. Be sure to stay at a safe distance.* Light the magnesium ribbon, and step aside. What sequence of reactions occurs? Results of each reaction? Examine the products when feasible. What are the advantages of aluminum-reduced iron over carbon-reduced iron? Disadvantages?

Observations: The Mg ribbon ignites the ignition mixture which then ignites the thermite. The molten material flows into the sand and solidifies. Breaking the solid material shows a glassy matrix which contains a solid pellet or pellets of Fe which may be detected by use of a magnet. The aluminum-reduced iron has no carbon impurity. The disadvantage is that it is not feasible to produce large quantities as can be produced in the blast furnace.

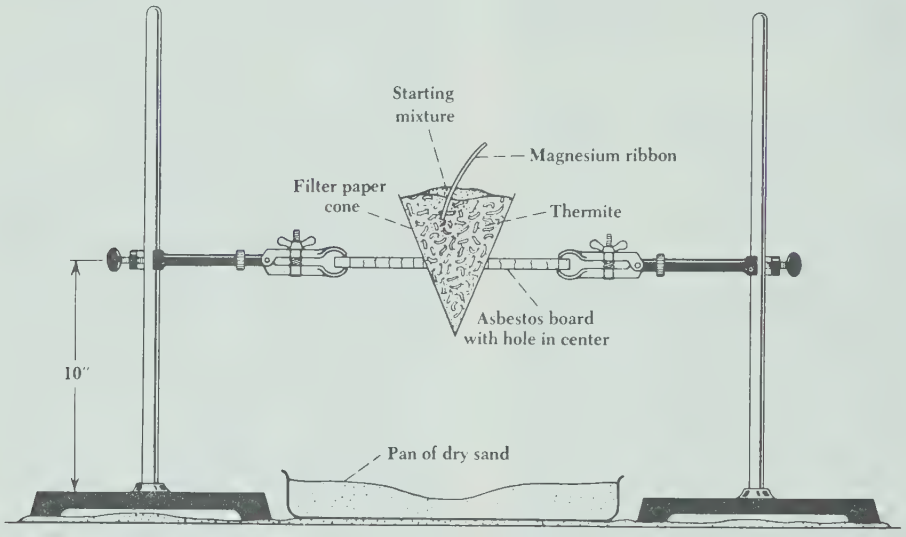

Fig. 54-1

EQUATIONS

1. $2Al + 3Hg_2(NO_3)_2 \rightarrow$ $2Al(NO_3)_3 + 6Hg(l)$
2. $2Al + 6HCl \rightarrow$ $2AlCl_3 + 3H_2(g)$
3. $2Al + 3H_2SO_4 \rightarrow$ $Al_2(SO_4)_3 + 3H_2(g)$
4. $Al + HNO_3 \rightarrow$ no perceptible reaction
5. $2Al(s) + 2NaOH + 2H_2O \rightarrow$ $2NaAlO_2 + 3H_2(g)$
6. $AlCl_3 + 3NH_3 + 3H_2O \rightarrow$ $Al(OH)_3(s) + 3NH_4Cl$
7. $H_3AlO_3 + 3NaOH \rightarrow$ $Na_3AlO_3 + 3H_2O$
8. $Al(OH)_3 + 3HCl \rightarrow$ $AlCl_3 + 3H_2O$
9. $2Al + Fe_2O_3 \rightarrow$ $2Fe + Al_2O_3$
10. $Al_2(SO_4)_3 + 3Na_2CO_3 + 6H_2O \rightarrow$ $2Al(OH)_3(s) + 3Na_2SO_4 + 3H_2CO_3$
11. $Al_2(SO_4)_3 + 3(NH_4)_2S + 6H_2O \rightarrow$ $2Al(OH)_3 + 3(NH_4)_2SO_4 + 3H_2S$

NAME ..... PERIOD ..... DATE .....
INSTRUCTOR'S COMMENTS .....

Experiment 55 Borax Bead Tests

PURPOSE: To learn how to identify metals by the color they impart to a borax bead.

APPARATUS: Burner and tubing; dish, evaporating; 5-cm length of No. 24 platinum wire, sealed in the end of a glass tube 10 cm long.

MATERIAL: Borax, powdered; chromium-(III) sulfate; cobalt(II) nitrate; iron(II) sulf fate; manganese(II) sulfate; nickel(II) chloride; unknown salt.

INTRODUCTION: Borax is hydrated sodium tetraborate, $Na_2B_4O_7 \cdot 10\ H_2O$. When it is heated strongly, it loses water of hydration and fuses to form a colorless glassy solid. A tiny trace of certain metallic compounds, when fused as oxides with such a borax glass, imparts a characteristic color which may be used to identify the metal. The colored bead formed in the loop at the end of a platinum wire is a metaborate of the metal.

PROCEDURE: 1. Make a borax bead as follows: Bend the tip of a platinum wire around the point of a lead pencil to form a loop about 3 mm in diameter. Heat the wire white-hot and then dip it into some powdered borax. Hold it in the oxidizing flame of your burner until the borax has melted to a clear glass. (See Fig. 55-1.) Avoid using too much borax as a large bead will simply drop off the wire. A certain amount of experimentation is necessary to get the best results.

Observations: Student answer .....

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2. Reheat the bead in the loop of wire and touch it, while hot, to a very small fragment (*not bigger than a tiny speck*) of cobalt(II) nitrate. Now heat the bead again in the oxidizing flame. The first fragment will probably be enough to impart a distinct color to the bead. If it does not do so, add another tiny speck of the cobalt(II) salt. The bead that forms should be colored, but clear and transparent. If it is black or opaque, too much cobalt(II) nitrate was used. In such an event, you must make a new bead and start over. To remove the bead from the wire, heat it strongly, hold it over an evaporating dish, and strike the hand holding the wire against the clenched fist of your other hand. Most of the bead will be dislodged and drop into the evaporating dish. Traces of the bead remaining can be removed by heating the wire and then plunging it into cold water. (**CAUTION:** *Do not dip the end of the glass handle in the water.* Why?) The shattered fragments of the bead can then be rubbed off between the fingers.

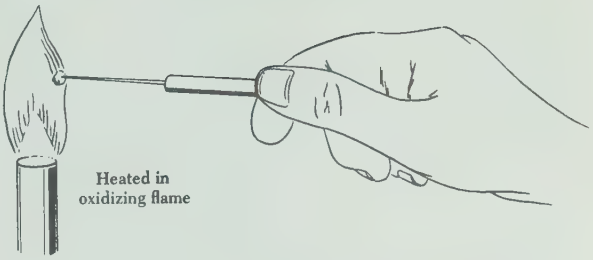

Fig. 55-1

Observations: The heated handle might crack in the water. .....

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3. Before making a new bead with a different metallic compound, clean the wire by making a borax bead and shaking it off into the evaporating dish. Repeat if necessary. The melted borax removes traces of the colored bead from the previous test.

Determine the color imparted to borax beads by traces of the following compounds: chromium(III) sulfate, manganese(II) sulfate, and nickel(II) chloride. Record your results in the Data Table.

Observations: Student answer

4. The color of a bead, colored by an iron compound, depends on whether the bead is heated in the oxidizing or in the reducing flame of your burner. Iron(II) metaborate has a different color from iron(III) metaborate. Add a trace of iron(II) sulfate to the borax bead, and then heat it in the oxidizing flame of your burner. Repeat, using iron(II) sulfate, but hold the bead in the reducing flame. (See Fig. 55-2.) Allow this second bead to cool in the unburned gas just above the top of the burner before you expose it to the air. Record the results in the Data Table as before.

Observations: Student answer

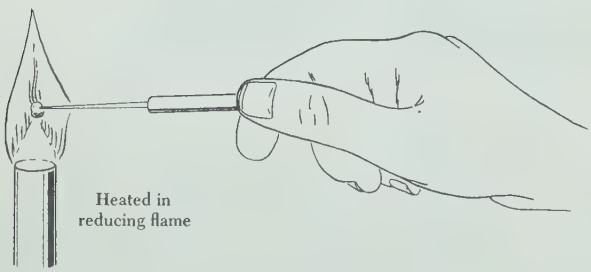

Fig. 55-2

5. Procure an unknown from your instructor and proceed to identify the metal it contains by the borax bead test. Record your results in the Data Table.

Observations: Student answer

DATA TABLE

Metal present	Bead color (when cool)
cobalt	blue
chromium	green
manganese	violet
nickel	red
iron (oxidizing flame)	brown-red
iron (reducing flame)	yellow
unknown	

The metal present in the unknown was . . .

QUESTIONS: Answer in complete statements.

1. Why is the iron-bead color different when formed in the oxidizing flame rather than in the reducing flame? The iron used contained the iron(II) ion. When the bead is formed in the oxidizing flame, the iron(II) is oxidized to iron(III) state. In the reducing flame, however, it remains in the lower oxidation state.

2. What coloring element would you suspect in pale-green glass? What would be the likely source? The coloring element is probably iron which was present in the sand used to make the glass.

Experiment 56

NAME ..... PERIOD ..... DATE .....

INSTRUCTOR'S COMMENTS .....

Separation of Metallic Ions

PURPOSE: To devise and carry out separations of metallic ions from certain suggested pairs.

APPARATUS: Graduated cylinder; test tubes.

MATERIAL: Litmus paper, red; acetic acid, glacial; ammonia-water solution, dilute (1:4); solutions of: aluminum chloride (0.1 *M*); copper(II) sulfate (0.1 *M*); iron(III) chloride (0.1 *M*); potassium hexacyanoferrate(II) (0.1 *M*); sodium hydroxide (3 *M*); zinc sulfate (0.1 *M*).

INTRODUCTION: Some metals have amphiprotic hydroxides while others do not. Certain transition metals form ammine complexes; others do not. In the metallurgy of aluminum it is necessary to remove iron from crude bauxite before electrolysis of the purified aluminum oxide. You will devise a procedure for simulating this process by separating iron from a mixture of aluminum and iron(III) salts. Other pairs of ions for which you will devise separation procedures include Cu^{++} and Fe^{+++}, Cu^{++} and Zn^{++}, and Zn^{++} and Al^{+++}. You are to use the reagents which are supplied. The series of preliminary experiments produce the results upon which your separation procedures are to be based. You may wish to check the results of these procedures against those in Experiments 49-51, and 54. It will be necessary to filter and wash precipitates to remove residual ion traces.

SUGGESTION: The instructor may assign one or more of the problems to individual students or student pairs.

PROCEDURE: 1. Iron(III) ion. To 5 ml of iron(III) chloride solution, add 1 ml of ammonia-water. Result? Now add ammonia-water until the mixture is alkaline to litmus. Result? Explain. Repeat with 5 ml of iron(III) chloride solution and sodium hydroxide solution. Result? Explain. Write the equations representative of your results.

Observations: A red-brown gelatinous precipitate of $Fe(OH)_3$ forms. This continues to be produced on addition of ammonia-water with no evidence of solution due to complex-ion formation. $Fe(OH)_3$ is also produced with NaOH solution. It does not dissolve in excess NaOH because it is not amphiprotic.

$FeCl_3 + 3NH_3 + 3H_2O \rightarrow Fe(OH)_3(s) + 3NH_4Cl$

2. *Aluminum ion.* To 5 ml of aluminum chloride solution, add 1 ml of ammonia-water. Result? What products are formed? Shake the test tube to flocculate the precipitate. Allow the precipitate to settle and pour off the supernatant liquid. Transfer half of the precipitate to a second test tube. To the precipitate in one test tube, add 1 ml of sodium hydroxide solution. Result? Explain. To the precipitate in the other test tube add ammonia-water until the mixture is alkaline to litmus. Result? Explain. Write the equations representative of your results.

Observations: A white precipitate of $Al(OH)_3$ forms. This dissolves in excess NaOH since $Al(OH)_3$ is amphiprotic, but does not dissolve in excess ammonia-water since it does not form a complex with NH_3.

$AlCl_3 + 3NH_3 + 3H_2O \rightarrow Al(OH)_3(s) + 3NH_4Cl$

$Al(OH)_3(s) + OH^- \rightarrow AlO_2^- + 2H_2O$

3. *Copper(II) ion.* To 5 ml of copper(II) sulfate solution, add 2 or 3 drops of ammonia-water. Result? Now add ammonia-water to excess (mixture alkaline to litmus). Result? Explain. Repeat with 5 ml of copper(II) sulfate solution and sodium hydroxide solution. Results? Explain. Write the equations representative of your results.

Acidify 5 ml of copper(II) sulfate solution by adding 3 drops of glacial acetic acid. Then add 5 drops of $K_4Fe(CN)_6$ solution. Result? Describe the precipitate. It may be considered to be $Cu_2Fe(CN)_6$. This is a reliable, qualitative test for copper(II) ions. This test may often be used to detect traces of copper.

Observations: A pale-blue precipitate of $Cu(OH)_2$ forms. This dissolves in excess ammonia-water and a dark blue solution results. NaOH produces a precipitate of $Cu(OH)_2$ which does not dissolve perceptibly in excess NaOH. The $Cu(OH)_2$ is not amphiprotic but does form the complex $Cu(NH_3)_4^{++}$ ion.

$CuSO_4 + 2OH^- \rightarrow Cu(OH)_2(s) + SO_4^=$

$Cu(OH)_2(s) + 4NH_3 + Cu(NH_3)_4^{++} + 2OH^-$

The red precipitate of $Cu_2Fe(CN)_6$ is produced.

4. Zinc ion. To 5 ml of zinc sulfate solution, add 2 or 3 drops of sodium hydroxide solution. Result? Shake the test tube and divide the contents into two parts. To one portion add sodium hydroxide solution to excess (alkaline to litmus). Result? Explain. To the other portion add ammonia-water to excess. Result? Explain.

Observations: A white precipitate of $Zn(OH)_2$ forms. It dissolves in excess NaOH since it is amphiprotic and also dissolves in excess ammonia-water due to the formation of the complex ion, $Zn(NH_3)_4^{++}$.

5. Separation of metallic ions from ion pairs. Devise and carry through separatory procedures for the ion-pairs listed below. Prepare the test mixtures by using equal volumes of their individual solutions. In sequential form, outline the procedures you used and found to be successful in the problem(s) assigned by the Instructor. Use the space below for your outline.

a. iron(III) and aluminum ions
b. iron(III) and copper(II) ions
c. copper(II) and zinc ions
d. zinc and aluminum ions

Experiment 57

NAME ..... PERIOD ..... DATE .....

INSTRUCTOR'S COMMENTS .....

Ammonia, the Ammonium Ion, and the Nitrate Ion

PURPOSE: To prepare ammonia, study its properties, and to differentiate between the ammonia molecule and the ammonium ion. To learn how to identify the nitrate ion.

APPARATUS: Balance, platform; beaker, 250 ml; 2 bottles, wide mouth; burner and tubing; clamp, buret; clamp, test tube; forceps; glass bend; glass plates; graduated cylinder; mortar and pestle; ring stand; 3 rubber stoppers, solid No. 2; rubber stopper, 1-hole No. 4; test tubes; test tube, Pyrex; wing top.

MATERIAL: Ammonium chloride; ammonium sulfate; calcium hydroxide; iron(II) sulfate; litmus papers, red and blue; wooden splints; ammonia-water solution, concentrated; hydrochloric acid, concentrated; sulfuric acid, concentrated; solutions of: potassium hydroxide (2.5 *M*); sodium hydroxide (2.5 *M*); sodium nitrate (0.5 *M*).

INTRODUCTION: Almost everyone is familiar with the penetrating odor of ammonia. Ammonia, NH_3, is a gas at ordinary temperatures and pressures. The ammonium ion, NH_4^+, is a radical with a positive charge of one which is never found alone, but always in conjunction with a negative ion. When a solution of a hydroxide reacts with an ammonium compound, the products are a salt, ammonia, and water. If any ammonium hydroxide molecules are formed at all, they do not persist as such, but immediately decompose into ammonia and water. Some of the ammonia escapes from the solution as a gas; as a result, such mixtures have the readily detected odor of ammonia.

PROCEDURE: 1. Put 0.5 g of dry ammonium chloride on one square of paper, and 0.5 g of calcium hydroxide on another square. Smell each of the chemicals in turn. Result? Then mix the chemicals, stirring them together. Put a small quantity of the mixed chemicals in the palm of one hand, and then rub your palms together for a half-minute. Now, *cautiously*, smell the mixture in your hand. Result? Hold a piece of wet, red litmus paper just above the chemical mixture in your palm. Result? Explain.

Observations: Neither solid has an odor. Rubbing the mixture together results in the perceptible odor of NH_3. Wet litmus paper turns blue in the vapor.

2. Repeat Part 1, using ammonium sulfate instead of ammonium chloride.

Observations: The same results take place as in 1.

3. Fit a large Pyrex test tube with a rubber stopper and L-tube as shown in Fig. 57-1. Gently mix 4 g of ammonium chloride and 3 g of calcium hydroxide in a mortar, and then add the mixture to the test tube. Spread the mixture in the test tube. Why? Clamp the test tube in the position shown for collecting the gas. The round end of the test tube should be slightly higher than the

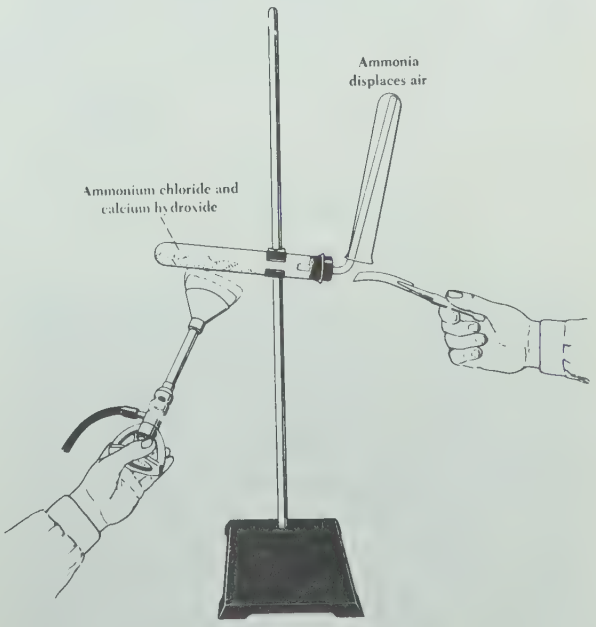

Fig. 57-1

stopper end. Why? (What products are formed?) Heat the test tube gently and collect three dry, small test tubes of the gas and stopper them. The color change of moist strips of red litmus paper held about two inches from the open end of a test tube will indicate when it is full of ammonia. What physical properties of ammonia have you observed in this preparation?

Observations: The mixture is spread along the lower bottom part of the test tube to insure even heating and easy escape of the NH_3 gas. The round end is higher to permit the escape of water which condenses. Ammonia is lighter than air and soluble in water.

4. Using the sample of ammonia in one small test tube, devise and conduct an experiment to determine the degree of solubility of ammonia in water. Describe your experiment and the results you obtain.

Observations: Student answer

5. Using the sample of ammonia in a second small test tube, devise and conduct an experiment to determine if ammonia burns and/or supports combustion. Describe your experiment, including the proper test tube position, and the results you obtain.

Observations: NH_3 neither burns in air nor supports combustion.

6. Adjust the generator so that ammonia can be passed into a small beaker half-full of water for a few minutes. Do not let the end of the tube touch the surface of the water. Why? Using an appropriate technique, test the solution formed with litmus paper. Result? Then pour 10 ml of the solution formed into a test tube. Boil the solution for several minutes, using a low burner flame. From time to time test the solution, using a proper method, to determine whether all the gas is expelled from solution by boiling. Result?

Observations: The very soluble NH_3 might cause the H_2O to be sucked back into the generator. Litmus paper turns blue. Boiling will remove most or all of the NH_3. The residue may be tested with litmus or phenolphthalein.

7. Investigate the possibility of a reaction between ammonium chloride and sodium hydroxide in solution. Determine the necessary conditions and the probable products of such a reaction. How could you identify the products? After devising a suitable technique, carry out the reaction, identifying the gaseous product. Describe your experimental method and your results. In a similar manner, investigate the possibility of a reaction between ammonium sulfate and potassium hydroxide in solution. Carry out the reaction, identifying the gaseous product. Describe your results.

Observations: You may heat a solid NH_4Cl with a solution of NaOH and detect the escaping NH_3 by odor and/or litmus paper reaction. It is also possible to use solid NaOH but care must be taken in handling the solid caustic NaOH. Ammonium sulfate and potassium hydroxide solution react to produce NH_3.

8. Add one gram of ammonium chloride crystals to a dry test tube. Using a low burner flame, heat the tube gently. At the same time, hold with forceps a strip of moist red litmus paper inside the mouth of the tube. Keep the litmus paper moist by adding a drop of water from a glass rod as needed. Look for first one, and then another, color change to occur in the litmus paper. Result? Why? What collects on the upper walls of the test tube? How did it get there?

Observations: The litmus paper may turn blue due to decomposition of the heated NH_4Cl which reforms in the cold upper part of the test tube and deposits as a solid on the walls of the test tube.

9. Using a low burner flame, carefully heat two wide-mouth bottles until they are moderately warm to the touch. Add three drops

of concentrated hydrochloric acid to one bottle and cover it with a glass plate. What happens to the concentrated hydrochloric acid when placed in a warm bottle? Take the other bottle to a distant corner of the laboratory and there add three drops of concentrated ammonia-water solution to it and cover with a glass plate. What happens to the concentrated ammonia-water solution? Bring the covered bottle containing ammonia to your work place and invert it over the bottle containing the hydrogen chloride. Remove the glass plates. Result? Explain.

Observations: The HCl vaporizes to yield HCl(g). Similarly, the heated ammonia-water liberates $NH_3(g)$. A white cloud of small solid particles of NH_4Cl results.

10. Prepare a solution of ammonium chloride by adding 0.5 g of this salt to 10 ml of water. Similarly prepare a solution of ammonium sulfate. Test each solution with red and blue litmus papers. Results? Explain the results in terms of the Brønsted acid-base concept.

Observations: Both solutions turn blue litmus to pink. The NH_4^+ ion is a weak acid and reacts with water to form H_3O^+ ion by hydrolysis.

11. Place 5 ml of freshly prepared iron(II) sulfate solution in a test tube. Add 2 ml of sodium nitrate solution. Mix the solutions, and then add 3 ml of concentrated sulfuric acid, holding the test tube in an inclined position so the acid will run down the side of the tube without mixing with the contents. The dense sulfuric acid sinks to the bottom of the tube. The colored layer formed where the two liquids meet serves as *test for a nitrate*. Repeat the test, using 1 nitric acid: 20 water (you prepare) instead of sodium nitrate solution. Result?

Observations: A brown ring forms with each nitrate tested.

EQUATIONS: Write ionic equations where appropriate.

1. Ammonium chloride and calcium hydroxide. $2NH_4Cl + Ca(OH)_2 \rightarrow CaCl_2 + 2NH_3(g) + 2H_2O$
2. Ammonium sulfate and calcium hydroxide. $(NH_4)_2SO_4(s) + Ca(OH)_2 \rightarrow CaSO_4(s) + 2NH_3(g) + 2H_2O(g)$
3. Ammonia and water. $NH_3 + H_2O \rightleftarrows NH_4^+ + OH^-$
4. Ammonium chloride and sodium hydroxide. $NH_4Cl + Na^+ + OH^- \rightarrow Na^+ Cl^- + NH_4^+ + OH^-$
5. Ammonium sulfate and potassium hydroxide. $(NH_4)_2SO_4 + 2KOH \rightarrow K_2SO_4 + 2NH_3(g) + 2H_2O$
6. Heating dry ammonium chloride. $NH_4Cl \rightarrow NH_3(g) + HCl(g)$
7. Ammonia and hydrogen chloride. $NH_3 + HCl \rightarrow NH_4Cl(s)$
8. Ammonium ion and water. $NH_4^+ + H_2O \rightleftarrows NH_3 + H_3O^+$

Experiment 58

NAME ..... PERIOD ..... DATE .....

INSTRUCTOR'S COMMENTS .....

Forms of Sulfur

PURPOSE: To prepare three solid allotropic forms of sulfur, and to study the physical and chemical properties of sulfur.

APPARATUS: Asbestos square; balance, platform; beaker, 250 ml; burner and tubing; clamp, test tube; dish, evaporating; forceps; funnel; graduated cylinder; magnifier; mortar and pestle; ring, iron; ring stand; spoon, deflagrating; test tube, Pyrex.

MATERIAL: Carbon disulfide; copper, strips of foil about 10 cm long and 0.5 cm wide; sulfur, flowers; sulfur, roll; zinc dust; asbestos paper; filter paper.

SUGGESTION: To avoid getting hot sulfur spilled on the laboratory table top, a large square of asbestos paper (or even heavy wrapping paper) may be used to cover it temporarily. A little cold water may be poured upon any burning sulfur which drops on the paper.

Since it is almost impossible to clean the test tubes used, the instructor may exchange clean test tubes for the ones used in this experiment and save the sulfur-encrusted ones for use with a later class or the class next year.

CAUTION: The vapor from carbon disulfide *is poisonous. It is very flammable, explosively so when mixed with air. Keep flames away from it.*

INTRODUCTION: Sulfur, like several other elements, exists in different allotropic modifications. Sulfur has three solid allotropic forms. Liquid allotropic forms of sulfur, of decidedly different fluidity, also exist.

PROCEDURE: 1. Powder a lump of roll sulfur, about the size of a small pea, and dissolve it in 8 ml of carbon disulfide in an evaporating dish. Stir with a glass rod to hasten solution. Let the liquid evaporate *spontaneously* in a good draft of air in a hood or on the sill of an open window. Use a magnifier to examine the crystals. Describe their shape and appearance. Which allotropic form of sulfur is this?

Observations: The pale-yellow crystals are rhombic in shape and are the rhombic allotropic form of sulfur.

2. Fill a test tube half-full of small pieces of sulfur, and melt the sulfur *at as low a temperature as possible.* During the heating, the liquid should not get much darker than a pale-straw color. Pour the molten sulfur into a folded dry filter paper cone in a funnel supported on a ring stand. Watch it as it cools. *As soon as* needle-like crystals grow across the surface, pour the excess liquid into a beaker of cold water. Unfold the filter paper *at once* and examine the crystals with a lens. Describe their shape and appearance and compare them with those obtained in Part 1. Which allotropic form of sulfur is this? Keep the crystals obtained in Part 2 for a few days and examine them closely again for any changes.

Observations: The crystals are needle-like in appearance. They are the monoclinic allotropic form of sulfur.

3. Fill the same test tube you used in Part 2 half-full of small pieces of sulfur and heat it, slowly at first. Note the color when the sulfur first melts, and also its fluidity. Observe any changes in color or fluidity that occur when the sulfur is heated more strongly. Its fluidity can be observed by holding the tube in a nearly horizontal position. Finally heat the sulfur to boiling, extinguish the burner flame, and pour the contents of the tube into a beaker of cold water. At this point, the sulfur may catch fire. Do not become excited, the sulfur will burn quietly. After emptying the test tube,

any sulfur on it which is still burning may be smothered with a towel. Remove the product from the beaker of water and examine it. Which allotropic form of sulfur is this? Keep it for a few days and examine it again.

Observations: When sulfur first melts it is a pale-yellow liquid. Further heating causes the sulfur to darken in color to amber and then to reddish-brown. It becomes more viscous at this stage. Further heating causes it to turn almost to black and it becomes fluid and starts to boil. The rubbery, plastic mass of sulfur that forms in the cold water is amorphous sulfur.

4. Burn a tiny piece of sulfur in an asbestos-lined deflagrating spoon. Note the color of the flame and the odor of the gas that is formed. **CAUTION:** See Fig. F-5 for the proper method of detecting the odor of a gas.

Observations: Sulfur burns with a purple flame producing SO_2 which has a suffocating choking odor.

5. Heat the test tube used in Part 3 until the sulfur that stuck to the walls of the tube begins to boil. Hold a piece of bright, untarnished copper foil with the forceps, and thrust it down into the vapor from the boiling sulfur. After a few moments, remove the foil and examine it. Result?

Observations: The copper foil flows with a red heat as the black copper(I) sulfide is produced.

6. Procure 0.5 g of zinc dust from your instructor. Mix it on an asbestos square with an equal bulk of flowers of sulfur. Heap it up into a conical pile, place it in the hood, and (**CAUTION**), holding the burner at arm's length, ignite it with the tip of the flame of your burner. Result?

Observations: The pile ignites forming a white mushroom shaped could and leaving a deposit of solid which is white to pale yellow in color. The product is ZnS.

QUESTIONS: 1. What changes in the structure of sulfur molecules occur as sulfur is raised from its melting point to its boiling point? The eight-membered sulfur molecules of solid sulfur melt to eight-membered rings of lambda-sulfur. As the temperature rises, these rings open, link together to form long chains of mu-sulfur, yet ultimately break up and boil to form S_8 sulfur vapor molecules.

2. What changes in structure does amorphous sulfur undergo when it stands for some time? The molecular chains gradually break up and form S_8 molecules of rhombic sulfur.

EQUATIONS

1. Reaction in Part 4. $S + O_2 \rightarrow SO_2(g)$
2. Reaction in Part 5. $2Cu + S \rightarrow Cu_2S$
3. Reaction in Part 6. $Zn + S \rightarrow ZnS$

Experiment 59

NAME. . . . . PERIOD . . . . . DATE. . . . . .

INSTRUCTOR'S COMMENTS . . . . . .

Hydrogen Sulfide and Qualitative Analysis

PURPOSE: To prepare hydrogen sulfide and study some of its properties. To show how hydrogen sulfide is used in qualitative analysis.

APPARATUS: 2 bottles, wide mouth; burner and tubing; clamp, buret; funnel; funnel tube; 2 glass bends; glass plate; graduated cylinder; jet tip; ring, iron; ring stand; rubber connector; rubber stopper, 2 hole No. 6; test tubes.

MATERIAL: Iron(II) sulfide; silver coin; filter paper; litmus papers, red and blue; ammonia water solution, concentrated; hydrochloric acid, concentrated; nitric acid, concentrated; solutions of: antimony(III) chloride (0.25 *M*); cadmium nitrate (0.25 *M*); copper(II) nitrate (0.25 *M*); hydrogen peroxide (3%); lead acetate (0.25 *M*); manganese(II) sulfate (0.25 *M*); methyl orange; zinc sulfate (0.25 *M*); stock solution of Cu^{++}, Mn^{++}, Zn^{++} (see Teachers' Edition). **CAUTION:** *Hydrogen sulfide is disagreeable and poisonous. Use the hood or work where there is a good draft. It is more satisfactory to do the experiment as a demonstration.*

INTRODUCTION: Hydrogen sulfide is usually prepared by the reaction between iron(II) sulfide and hydrochloric or sulfuric acid. Hydrogen sulfide forms the weak diprotic hydrosulfuric acid when dissolved in water. Saturated solutions of hydrogen sulfide are approximately 0.1 *M* with respect to H_2S. Combining the $K_{ion\ 1}$ with the $K_{ion\ 2}$ for this solution established the following relationship:

$$[S^{--}] = \frac{6.8 \times 10^{-24}}{[H_3O^+]^2}$$

This indicates that the sulfide ion concentration in hydrogen sulfide solutions varies inversely as the square of the hydronium ion concentration.

In qualitative analysis certain metallic ions may be separated from each other by controlling the hydronium ion concentration at which saturated hydrogen sulfide is added. One group, which includes Hg^{++}, Pb^{++}, Bi^{+++}, Cu^{++}, and Cd^{++} is precipitated in a solution which is acid to methyl orange. The acid color of methyl orange is red at a pH of 3.1. A second group of ions, whose sulfides do not precipitate at this pH, includes Fe^{+++}, Mn^{++}, Al^{+++}, Cr^{+++}, Ni^{++}, Co^{++} and Zn^{++}. Thier solubility products are relatively large compared to those of the first group. If all of these ions are included in the acidified sample originally treated with hydrogen sulfide, certain ones may be precipitated as hydroxides by making the filtrate alkaline with ammonia-water. These hydroxides have characteristic colors. Ni^{++}, Co^{++}, and Zn^{++} do not precipitate as hydroxides because they form complex ions such as $Zn(NH_3)_4^{++}$. The hydroxides are removed by filtration and the alkaline filtrate is again treated with hydrogen sulfide. The sulfides of these three ions do precipitate and may be identified by their color and supplementary identification procedures.

PROCEDURE: 1. Set up the apparatus as shown in Fig. 59-1 to collect the gas by air displacement. Why? Put 2 or 3 small lumps of iron(II) sulfide into the generator, and add enough water through the funnel tube to cover its lower end. Then add concentrated hydrochloric acid, a little at a

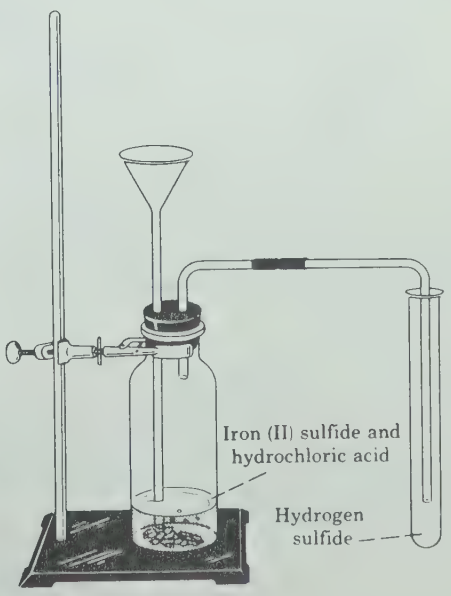

Fig. 59-1

time, to maintain a steady evolution of the gas. What physical properties of hydrogen sulfide do you observe?

Observations:

Hydrogen sulfide is soluble in water and is therefore collected by air displacement. The gas is heavier than air and has the odor of decayed eggs.

2. Collect a test tube of the gas. Hold it mouth upward, and ignite it. Note the odor of the burning gas (refer to Fig. F-5), and observe carefully the deposit on the walls of the tube.

Observations:

The burning H_2S produces a gas with a choking odor, SO_2. Some yellow sulfur deposits on the walls of the test tube.

3. Replace the glass bend at the end of the delivery tubing with a jet tip and light the gas at the end of the jet tip. Try to identify the products of combustion in this case. Are they the same as in Part 2 or different? Explain.

Observations:

The gas burns with a blue flame producing SO_2 but gives no evidence of sulfur production. The excess of air (oxygen) produces the more complete combustion.

4. Replace the jet tip with the glass bend. Pour 50 ml of water into a wide-mouth bottle and bubble hydrogen sulfide through the water for about 5 minutes. Using a proper technique, determine the effect of this solution on red and blue litmus test strips. Result? Use a stirring rod to transfer a drop of the hydrogen sulfide solution to a silver coin. Result? The reaction that occurs serves as a test for a soluble sulfide such as hydrogen sulfide. Go on to Part 5, but save the solution you just prepared for use in Part 7 and Part 8.

Observations:

The solution of H_2S turns blue litmus to red forming the weak hydrosulfuric acid. The silver coin turns black due to tarnishing and formation of black Ag_2S.

5. Permit the gas from the generator to bubble through 5 ml of hydrogen peroxide in a test tube from 3 to 5 minutes or until you notice a decided change. Result? What reaction occurred? Pour the contents of the test tube into a filter paper cone in a funnel supported by a ring and ring stand. What is the appearance of the filtrate? What explanation can you offer?

Observations:

A white colloidal suspension of sulfur forms. The H_2S is a reducing agent undergoing oxidation while the peroxide is reduced to oxide. Since the filtrate is cloudy, the sulfur is in colloidal suspension

6. Moisten a strip of filter paper with lead(II) acetate solution (or use a prepared test strip) and hold it at the end of the delivery tube from the hydrogen sulfide generator. Result? This reaction is a common test for hydrogen sulfide.

Observations:

The brown-black deposit of PbS forms.

Remove the stopper from the generator and quickly pour the excess acid from the iron(II) sulfide. Flush the acid down the sink with water. Rinse the iron(II) sulfide remaining in the generator with water. Place the well-rinsed iron(II) sulfide in the designated container.

7. Pour about 5 ml of hydrogen sulfide solution from Part 4 into each of five test tubes. To the first one, add 1 ml of copper(II) nitrate solution; to the second, 1 ml of antimony(III) chloride solution; to the third, 1 ml of cadmium nitrate solution; to the fourth, 1 ml of zinc sulfate solution; and to the fifth, 1 ml of manganese(II) sulfate solution. Results? Little or no precipitate will appear in the case of zinc and manganese, since their sulfides will not form unless the solution is alkaline. To the test tubes containing Zn^{++} and Mn^{++}, add two or three drops of ammonia-water solution. Results? What colors are the products?

Observations:

The sulfides first produced are black CuS, orange Sb_2S_3, yellow CdS. A white precipitate of ZnS and a pink precipitate of MnS may form initially but after the addition of ammonia-water, much greater precipitation takes place. ZnS and MnS precipitate in alkaline solution.

8. *a.* To 1 ml of the mixture of Cu^{++}, Zn^{++}, and Mn^{++} solution, add nitric acid dropwise until the solution tests acid with methyl orange. Add 5 ml of the hydrogen sulfide solution from Part 4. Result? Explain. Filter to remove the precipitate. Save the filtrate for *b.* below.

b. To the filtrate, add ammonia-water until the mixture tests alkaline to litmus paper. Result? Explain. Filter. Save the filtrate for *c.*

c. To the filtrate, add 5 ml of the hydrogen sulfide solution from Part 4. Result? Explain.

Observations:

a. Black CuS is precipitated from the acid solution.

b. Some precipitation of ZnS and MnS may occur upon addition of ammonia-water.

c. Addition of H_2S to the filtrate (alkaline) produces ZnS and MnS precipitates because the initial precipitation of CuS may have removed most of the sulfide ion. These two sulfides have much larger solubility-product constants than the CuS which is precipitated from acid solution.

EQUATIONS: Write ionic equations where appropriate.

1. Preparation of hydrogen sulfide. $FeS + 2HCl \rightarrow FeCl_2 + H_2S(g)$
2. Complete combustion of hydrogen sulfide. $2H_2S + 3O_2 \rightarrow 2H_2O + 2SO_2(g)$
3. Hydrogen sulfide and silver in the presence of air. $4Ag + 2H_2S + O_2 \rightarrow Ag_2S(s) + 2H_2O$
4. Reaction in Part 5. $H_2O_2 + H_2S \rightarrow 2H_2O + S(s)$
5. Reaction in Part 6. $Pb(C_2H_3O_2)_2 + H_2S \rightarrow PbS(s) + 2HC_2H_3O_2$
6. First three reactions in Part 7. $Cu(NO_3)_2 + H_2S \rightarrow CuS(s) + 2HNO_3$

$2SbCl_3 + 3H_2S \rightarrow As_2S_3(s) + 6HCl$

$Cd(NO_3)_2 + H_2S \rightarrow CdS(s) + 2HNO_3$

7. Reactions for separation of ions in Part 8. *a.* $Cu^{++} + S^{--} \rightarrow CuS(s)$

b. $Mn^{++} + 2OH^- \rightarrow Mn(OH)_2(s)$ $\quad Zn^{++} + 4NH_3 \rightarrow Zn(NH_3)_4{}^{++}$

c. $Zn(NH_3)_4{}^{++} + S^{=} \rightarrow ZnS(s) + 4NH^3$

Experiment 60

NAME . . . . . PERIOD . . . . . DATE . . . . . .

INSTRUCTOR'S COMMENTS . . . . . .

Sulfite and Sulfate Ions

PURPOSE: To show how the sulfite and sulfate ions may be identified. To study the properties of the acids and salts of these ions.

APPARATUS: Balance, platform; bottle, wide mouth; burner and tubing; clamp, buret; clamp, test tube; funnel; funnel tube; 3 glass bends; glass plate; graduated cylinder; ring, iron; ring stand; rubber connector; rubber stoppers, 1-hole No. 4, 2-hole No. 4; test tubes; 2 test tubes, large, Pyrex.

MATERIAL: Magnesium ribbon; sodium hydrogen sulfite; zinc, mossy; filter paper; litmus papers, red and blue; hydrochloric acid, concentrated; hydrochloric acid, dilute; solutions of barium chloride (0.25 *M*); potassium permanganate (0.1 *M*); sodium sulfate (0.1 *M*); sodium sulfite (0.1 *M*).

ADDITIONAL MATERIAL FOR INSTRUCTOR'S EXPERIMENT: Copper shot; sulfuric acid, concentrated; solution of potassium permanganate (0.1 *M*).

INTRODUCTION: The oxidation states of sulfur are -2, $+4$, and $+6$. It has an oxidation state of $+4$ in sulfur dioxide, sulfurous acid, and in the sulfite ion. It has an oxidation state of $+6$ in sulfuric acid and in the sulfate ion. Sulfuric acid is a powerful oxidizing agent when hot and concentrated. Sulfurous acid and sulfites in acid solution are reducing agents. In this experiment you will perform tests which will enable you to distinguish between the sulfite and sulfate ions. You will also study oxidation-reactions in which sulfur undergoes changes in its oxidation state.

PROCEDURE: 1. *Tests for the SO_4^{--} and SO_3^{--} ions.* *a.* To 4 ml of sodium sulfate solution, add 5 or 6 drops of dilute hydrochloric acid. Result? Add 2 ml of barium chloride solution to the mixture. Result? Identify the products.

Observations: Na_2SO_4 does not dissolve in HCl. Barium chloride addition produces a white precipitate of $BaSO_4$ along with soluble NaCl.

b. To 5 ml of sodium sulfite solution, add 5 or 6 drops of dilute hydrochloric acid. Result? Investigate the odor of the product by using the wafting motion (See Fig. F-5). Add 2 ml of barium chloride solution to the mixture. Results?

Observations: The suffocating, choking odor identifies the gas as SO_2. Addition of barium chloride does not produce a precipitate.

c. To 5 ml of sodium sulfite solution add 2 ml of barium chloride solution. Result? Explain. Add 5 or 6 drops of dilute hydrochloric acid to the mixture and shake the test tube. Result? Explain. Why is hydrochloric acid necessary in performing the test for these ions?

Observations: A white precipitate of $BaSO_3$ is produced. Addition of the HCl causes the precipitate of $BaSO_3$ to dissolve since the HCl causes the liberation of SO_2 with the decomposition of the $BaSO_3$. HCl must be used in order to distinguish the precipitates, $BaSO_3$ being soluble and $BaSO_4$ being insoluble.

d. Test 5 ml portions of the solutions of sodium sulfate and sodium sulfite respectively with red and blue litmus papers. Results? Explain. What do these results indicate about the relative strengths of the corresponding acids?

Observations: The Na_2SO_3 solution turns red litmus to blue while Na_2SO_4 does not affect litmus. The sulfurous acid is a weak acid and the sulfite ion undergoes hydrolysis. Sulfuric acid is a strong acid.

2. *Sulfur dioxide.* Set up the apparatus as shown in Fig. 60-1 to collect the gas by air displacement. Put 2 grams of sodium hydrogen sulfite into the generator. Place a piece of red and a piece of blue litmus paper (moistened) across the open end of a test tube and put the end of the delivery tube in the test tube. Add 2 ml of dilute hydrochloric acid through the funnel tube of the generator. As soon as the litmus paper turns color, remove the delivery tube from test test tube and place it in 20 ml of water in a wide-mouth bottle. What did the litmus paper result indicate about sulfur dioxide? Add 3 ml more hydrochloric acid through the funnel tube. When gas evolution stops, remove the delivery tube from the water

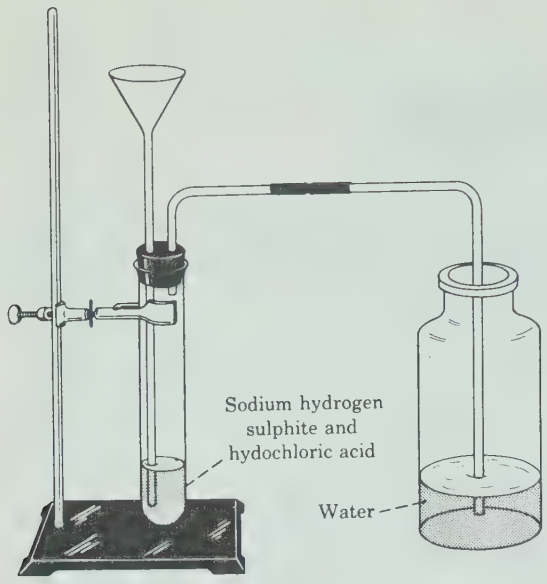

Fig. 60-1

in the bottle. Select the test(s) from Part 1 required to identify the anion present in 5 ml of this solution. Perform the selected test(s). Results? Explain.

To another 5 ml portion of the solution add a piece of zinc. Result? If no perceptible reaction occurs, decant the solution into another test tube. Add a 1 cm piece of magnesium ribbon to the solution. Result? What do these tests indicate about the nature of the solution?

Observations: The blue litmus paper turning red indicates that SO_2 is an acid anhydride. Adding $BaCl_2$ solution produces a white precipitate which dissolves upon addition of hydrochloric acid. This result identifies the SO_3^{--} ion as being present in the solution. Zinc may produce very little if any gas but magnesium produces much greater gas(H_2) evolution. These tests indicate the H_2SO_3 is a weak acid.

3. *Oxidation and reduction.* Potassium permanganate ($KMnO_4$) in acid solution is a vigorous oxidizing agent. In the presence of a reducing agent, the manganese in the purple permanganate ions, MnO_4^-, is reduced to the almost colorless manganese(II) ions, Mn^{++}. To the remaining 10 ml of water solution of sulfur dioxide from Part 2, add 2 ml of concentrated hydrochloric acid. Then add potassium permanganate solution dropwise until the last drop just produces a faintly pink solution. If the solution is not clear, filter it. Test 5 ml of the filtrate for the presence of SO_4^{--} ions. Result? What change occurred in the ions present in the solution of sulfur dioxide? What action did the sulfur in these ions have on the manganese in the permanganate ions? Is the sulfur in these ions an oxidizing or a reducing agent?

Observations: The filtrate gives a positive test for SO_4^{--} ions. The sulfite ion was oxidized to the sulfate ion while the manganese was reduced from an oxidation state of +7 in $KMnO_4$ to an oxidation state of +2 in Mn^{++}. The sulfur in the SO_3^{--} ions is a reducing agent.

4. INSTRUCTOR'S EXPERIMENT. Heat 2 g of copper shot with 2 ml of concentrated sulfuric acid in a test tube generator. Collect several test tubes of the gas and have students test its odor, reaction with litmus, and reaction with dilute potassium permanganate solution. Results? Explain. What process did the sulfur in the sulfuric acid undergo?

Observations: The gas produced has the choking, suffocating odor of SO_2; turns blue litmus to red; and reduces the purple permanganate to almost colorless Mn^{++}. The sulfur in the H_2SO_4 was reduced. Hot, concentrated H_2SO_4 is an oxidizing agent.

EQUATIONS: Write ionic equations where appropriate.

1. Test for sulfate ion. $SO_4^{--} + Ba^{++} \rightarrow BaSO_4(s)$
2. Reaction of sulfite ion with water. $SO_3^{--} + H_2O \rightarrow HSO_3^- + OH^-$
3. Action of hydrochloric acid and sodium sulfite. $Na_2SO_3 + 2HCl \rightarrow NaCl + H_2O + SO_2(g)$
4. Action of hydrochloric acid and sodium hydrogen sulfite. $NaHSO_3 + HCl \rightarrow NaCl + H_2O + SO_2(g)$
5. Action of magnesium with hydronium ion. $Mg + 2H_3O^+ \rightarrow Mg^{++} + 2H_2O + H_2(g)$
6. Action of sulfur dioxide and water. $SO_2 + H_2O \rightleftarrows H_2SO_3$

NAME. . . . . PERIOD . . . . . DATE . . . . . .
INSTRUCTOR'S COMMENTS . . . . . .

Experiment 61 Preparation and Properties of Chlorine

PURPOSE: To prepare chlorine and study its properties.

APPARATUS: Balance, platform; beaker, 250 ml; 6 bottles, wide mouth; burner and tubing; 2 clamps, buret; flask, Erlenmeyer; funnel tube; generator with delivery tube and bent jet tube; glass bends; 4 glass plates; graduated cylinder; ring, iron; 2 ring stands; 2 rubber stoppers; 2-hole No. 4; 4 rubber stoppers, 2-hole No. 6; spoon, deflagrating; test tubes; wire gauze.

MATERIAL: Antimony, powdered; bleaching powder, Ca(ClO)Cl; lead dioxide; manganese dioxide, pellets; manganese dioxide, powdered; potassium dichromate; potassium permanganate; zinc, mossy; candle; cotton cloth, colored; litmus papers, red and blue; paper, printed; paper, white; hydrochloric acid, concentrated; sulfuric acid, dilute; solution of sodium thiosulfate (0.25 *M*).

INTRODUCTION: Chlorine is prepared by the oxidation of the chloride ion. **CAUTION:** *Since chlorine is poisonous you will prepare very small amounts of it by using a variety of oxidizing agents.* A convenient laboratory preparation is the oxidation of hydrochloric acid with manganese dioxide. Excess chlorine may be bubbled through sodium thiosulfate solution which reduces chlorine to chloride ions, thus preventing the escape of chlorine into the room.

SUGGESTION: It is suggested that the preparation and collection of chlorine be performed by the instructor at the hood. The series of test tube preparations may be performed by the students.

PROCEDURE: 1. Set up the apparatus in the hood as shown in Fig. 61-1. Add 15 g of powdered manganese dioxide to the flask. and then pour 40 ml of concentrated hydrochloric acid through the funnel tube. The flask must be heated *very gently* to avoid too rapid production of chlorine. If a sheet of white paper is held behind the bottles, the color will indicate when they are full of the gas. What is the purpose of the HCl? Of the MnO_2? Why is chlorine led into the bottom of each collecting bottle?

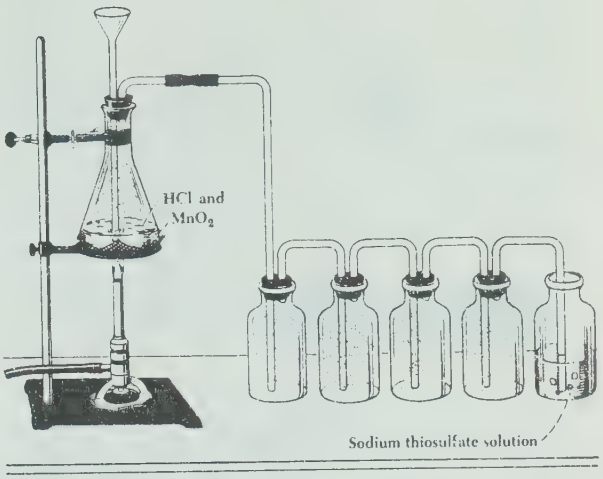

Fig. 61-1

Observations: The HCl is the source of the Cl_2. The MnO_2 is the oxidizing agent. Chlorine is heavier than air which is displaced upward as the Cl_2 fills the bottom of the bottle.

2. When the bottles are full of chlorine, disconnect the apparatus and leave the generator in the hood. Cover each bottle of chlorine with a glass plate.

3. To one of the bottles of chlorine, add 20 ml of water, and quickly cover with a glass plate or solid rubber stopper. Shake the bottle vigorously. Result? Note the color of the water. To what is this color due? Test the effect of chlorine water on strips of red and blue litmus papers. Result? Try the effect of chlorine water on a small square of colored cotton cloth. See if the chlorine water will bleach the printing on a small square of newspaper, or a piece of book paper. Explain any differences in the effectiveness of the bleaching action you observe.

Observations: The water solution appears to be faintly yellow-green. It is formed when Cl_2 is shaken with water. The chlorine water bleaches both the red and blue litmus papers. It does not bleach the printing ink of newspaper since the carbon in the ink is not oxidized.

4. Set up a hydrogen generator fitted with a glass jet tube which can be lowered into a bottle of chlorine as in Fig. 61-2. **CAUTION:** *Do not light the hydrogen until you have determined that all of the air has been*

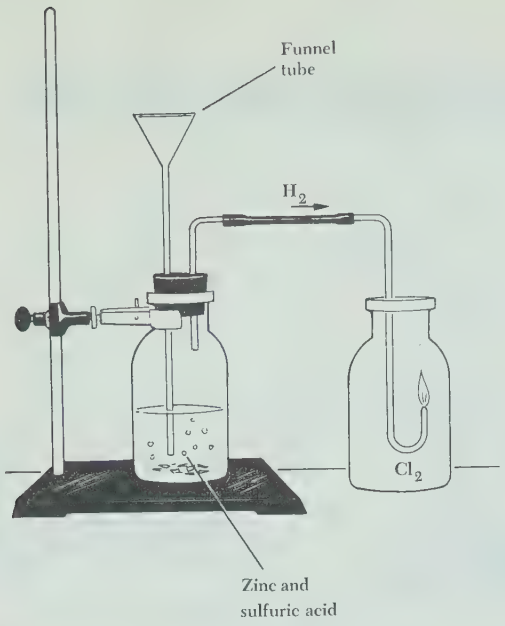

Fig. 64-2

expelled from the generator. To determine this, fill a test tube with the gas by water displacement and carry it mouth downward to a flame. If it burns with a slight explosion, or "barks" it is still impure. Collect another test tube and test as before. If the hydrogen is pure, it will burn quietly at the mouth of the test tube. The jet tube is then ignited by bringing this test tube, with hydrogen burning at its mouth, to the jet tip. Lower the jet tube of burning hydrogen into a bottle of chlorine. Does the hydrogen continue to burn in the chlorine? A moist strip of blue litmus paper may help you to identify the product. What is unusual about this reaction?

Observations: The hydrogen continues to burn in the chlorine. Moist blue litmus paper turns red in the HCl produced provided all the Cl_2 has been combined with H_2. The fuming characteristic of the action of HCl with moist air may take place around the moist litmus paper.

5. Candle wax is composed of hydrocarbons. Lower a lighted candle, mounted in a deflagrating spoon, into a bottle of chlorine. Result? Identify the products of combustion. Explain.

Observations: The candle continues to burn with a deep red and sooty flame depositing carbon. Litmus paper will turn red in the vapors of HCl produced. This will also fume, if your breath is blown across the top of the bottle.

6. Sprinkle some powdered antimony into a bottle of chlorine. Result? What is the product?

Observations: The antimony emits a shower of sparks forming a white cloud and a deposit of $SbCl_3$.

7. Half fill a 250-ml beaker with water and place it on a wire gauze mounted on a large ring on a ring stand. Heat thew ater to boiling to use as a hot water bath. In separate test tubes add 5 drops of concentrated hydrochloric acid to each of the following: (a) one pellet of MnO_2; (b) two grains of $KMnO_4$; (c) two crystals of $K_2Cr_2O_7$; (d) an amount of PbO_2 equal in bulk to a match head; (e) an amount of Ca(ClO)Cl equal in bulk to a match head. Warm each in turn in the boiling water. Observe the color of the gas produced against a white paper background. Then suspend a piece of moistened blue litmus paper in the test tube. Report the results.

Observations: In each case, Cl_2 is produced and is identified by the greenish-yellow color and the bleaching of the litmus paper.

EQUATIONS

1. $MnO_2 + 4HCl \rightarrow MnCl_2 + 2H_2O + Cl_2(g)$
2. $H_2O + Cl_2 \rightarrow HCl + HClO$
3. $H_2 + Cl_2 \rightarrow 2HCl$
4. $2Sb + 3Cl_2 \rightarrow 2SbCl_3$
5. $2KMnO_4 + 16HCl \rightarrow 2KCl + 2MnCl_2 + 8H_2O + 5Cl_2(g)$
6. $K_2Cr_2O_7 + 14HCl \rightarrow 2KCl + 2CrCl_3 + 7H_2O + 3Cl_2(g)$
7. $PbO_2 + 4HCl \rightarrow PbCl_2 + 2H_2O + Cl_2(g)$
8. $Ca(ClO)Cl + 2HCl \rightarrow CaCl_2 + H_2O + Cl_2(g)$

Experiment 62

NAME. . . . . PERIOD . . . . . DATE . . . . . .

INSTRUCTOR'S COMMENTS. . . . . .

Preparation and Properties of Iodine

PURPOSE: To prepare iodine and to study its properties.

APPARATUS: Balance, platform; burner and tubing; clamp, buret; funnel, separatory; glass plate; graduated cylinder; ring iron; ring stand; rubber band, heavy; test tubes; test tube, large, Pyrex.

MATERIAL: Alcohol, denatured; carbon tetrachloride; iodine, crystals; manganese dioxide, powdered; potassium bromide; potassium iodide, powdered; wooden splints; phosphoric acid, concentrated; solutions of: bromine water; chlorine water; potassium bromide (0.5 *M*); potassium iodide (0.5 *M*).

INTRODUCTION: Iodine is an active, solid, nonmetallic element which forms a colored vapor when heated. It is prepared from potassium iodide, manganese dioxide, and sulfuric (or phosphoric) acid. In this experiment we will use phosphoric acid because it yields no undesirable by-products. Iodine is less active chemically than chlorine or bromine, so it may be displaced from its compounds by either of these halogens. **CAUTION:** *Carbon tetrachloride vapor is poisonous. Do not inhale the vapor.* The laboratory should be well ventilated while carbon tetrachloride is being used. Perchlorethylene may be used instead.

PROCEDURE: 1. Mix 1 g of powdered potassium iodide with an equal bulk of powdered manganese dioxide and transfer the mixture to a Pyrex test tube. Wind a heavy rubber band around the outside lip of a smaller test tube so that this test tube will fit into the upper part of the larger generator test tube and be supported by the rubber band. Fill this smaller test tube with water. Pour 1 ml of concentrated phosphoric acid into the generator test tube and place the smaller tube inside. Heat the contents of the larger test tube *gently*. Note the color of any vapor produced in the tube. Heat for several minutes and permit the assembly to cool. Remove the smaller test tube and pour out the water. Note the color of the iodine crystals which have collected on the outside of this small test tube. What physical process does the deposition of these crystals represent?

Observations: The violet vapor of I_2 is produced and steel-gray crystals of I_2 adhere to the outside of the small test tube. These crystals were produced by sublimation.

2. With a wooden splint, scrape onto a glass plate a few iodine crystals from the test tube. Add a crystals or two of iodine to each of four test tubes. Compare the solubility of iodine in water, alcohol, carbon tetrachloride, and potassium iodide solutions. Note the color of each solution.

Observations: Iodine is practically insoluble in water but does dissolve in a solution of potassium iodide giving a brown solution. It forms a dark-brown solution in alcohol and a purple color in carbon tetrachloride.

3. Add 3 ml of carbon tetrachloride to a test tube half-full of water, and shake the tube vigorously. What happens when you stop shaking? What term is applied to such liquids? What happens to the carbon tetrachloride? Is it more dense or less dense than water?

Add 3 ml of carbon tetrachloride to 10 ml of bromine water and shake thoroughly. Observe by the depth of color whether bromine is more soluble in water, or in carbon tetrachloride. Recall from Part 2 whether iodine is more soluble in water or in carbon tetrachloride.

Observations: Two layers of liquids form, since carbon tetrachloride and water are immiscible. The heavier layer of CCl_4 is on the bottom. Bromine is much more soluble in CCl_4 since, on shaking the water solution with CCl_4, the colorless CCl_4 develops a deep orange-red color while most of the bromine has left the water, leaving the water solution faintly colored.

4. Dissolve one or two small crystals of potassium bromide in 10 ml of water. Add 3 ml of carbon tetrachloride and shake thoroughly. Do bromide ions color carbon tetrachloride? Now add 5 ml of chlorine water and again shake the test tube thoroughly. The carbon tetrachloride is merely a solvent and does not enter into the chemical reaction. By its color, you should be able to judge what did happen. Explain your result in terms of the halogen activity series. Similarly perform appropriate tests to show the reaction between iodide ions and chlorine molecules and the reaction between iodide ions and bromine molecules. Describe your tests, results, and reactions which occurred.

Observations:

Bromide ions do not color carbon tetrachloride. Addition of chlorine water causes the water layer to become orange-red. On shaking with the CCl_4, the characteristic deep orange-red color is concentrated in the CCl_4.

Chlorine is more active than bromine and replaces it from bromide solutions.

Both Cl_2 and Br_2 will replace iodine in iodide solutions producing the purple color with added CCl_4. These results indicate that the order of activity is $Cl_2 > Br_2 > I_2$.

5. Place 1 ml of potassium bromide solution, 1 ml of potassium iodide solution, and 10 ml of distilled water in a separatory funnel. Add 1 ml of carbon tetrachloride and 1 drop of chlorine water. Stopper the separatory funnel and shake it. Allow the liquid layers to separate. What is the color of the carbon tetrachloride layer? What does this indicate? Remove the stopper from the funnel and draw off the carbon tetrachloride layer into a test tube. Add 1 ml of carbon tetrachloride and 1 drop of chlorine water again to the liquid in the separatory funnel, stopper the funnel, and shake. Allow the liquids to separate. Result? Unstopper the funnel and draw off the carbon tetrachloride into a second test tube. Repeat the procedure until there is a significant change in the color of the carbon tetrachloride layer. What reaction is now occurring? Why?

Observations:

The CCl_4 layer is violet. The iodide ion has been changed (oxidized) to I_2. A number of additions of CCl_4 and chlorine water produces violet color in CCl_4 layer. There may be one addition finally which produces a colorless CCl_4 layer possibly due to oxidation of residual I^- to I_2 and to the colorless IO_3^- ion. Finally the orange-red (faint amber) color in the CCl_4 layer results. This indicates that the Br^- ion is being oxidized to Br_2.

QUESTIONS: 1. What is the meaning of the term, "Resublimed Iodine," on a bottle of iodine crystals?

The iodine crystals in the bottle have been purified by sublimation more than once.

2. How do you account for the difference in the color of solutions of iodine in the solvents you tested?

Iodine is so slightly soluble in water that it does not color the water. The violet color of iodine in carbon tetrachloride is that of molecules of iodine. The yellow-brown color of iodine in alcohol is believed to be due to the shifted position of iodine molecule electrons when they are near alcohol molecules. The brown color of iodine-potassium iodide solutions is characteristic of the I_3^- ion.

3. What do you conclude about the relative chemical activities of chlorine, bromine, and iodine?

Chlorine is more active than bromine and iodine. Bromine is more active than iodine.

4. If you were given a solution suspected of containing both bromide and iodide ions, how could you detect the presence (or absence) of each? Add a small quantity of carbon tetrachloride to the solution and a drop of chlorine water. Shake. A violet coloration of the carbon tetrachloride indicates the presence of iodide ion in the original solution. An orange coloration of the carbon tetrachloride layer indicates the presence of bromide ion and the absence of iodide ion in the original solution. If iodide ions are present, extract them by the use of small portions of carbon tetrachloride and dropwise additions of chlorine water. If both iodide and bromide ions are present, the orange coloration characteristic of bromine will appear in the carbon tetrachloride layer after all the ions have been removed from the solution as iodine.

EQUATIONS: Write ionic equations where appropriate.

1. Preparation of iodine. $2KI + MnO_2 + 2H_2SO_4 \rightarrow K_2SO_4 + MnSO_4 + 2H_2O + I_2(g)$
2. Replacement of bromine by chlorine. $2\,Br^- + Cl_2 \rightarrow 2\,Cl^- + Br_2$
3. Replacement of iodine by chlorine. $2\,I^- + Cl_2 \rightarrow 2\,Cl^- + I_2$
4. Replacement of iodine by bromine. $2\,I^- + Br_2 \rightarrow 2\,Br^- + I_2$

Experiment 63

NAME. . . . . PERIOD . . . . . DATE . . . . . .

INSTRUCTOR'S COMMENTS . . . . . .

Halide Ions

PURPOSE: To develop a series of tests which will identify the halogenide ions, F^-, Cl^-, Br^-, I^-.

APPARATUS: Graduated cylinder; stoppers, cork, for test tubes; test tubes.

MATERIAL: Carbon tetrachloride; litmus paper, blue; ammonia-water solution, dilute (1:4); nitric acid, dilute (1:5); solutions of: bromine water; calcium nitrate (0.5 *M*); chlorine water; potassium bromide (0.2 *M*); potassium chlorate (0.1 *M*), potassium iodide (0.2 *M*); silver nitrate (0.1 *M*); sodium chloride (0.1 *M*); sodium fluoride (0.1 *M*).

CAUTION: *Carbon tetrachloride vapor is poisonous. Do not inhale the vapor.* The laboratory should be well ventilated while carbon tetrachloride is being used. Perchloroethylene may be used instead.

INTRODUCTION: The principle oxidation state of the halogens is −1. With the exception of fluorine, they may exist in other oxidation states. The specific tests which you will develop in this experiment involve the production of recognizable precipitates, oxidation-reduction reactions involving the use of halogens as oxidizing agents, and the production of complex ions. Refer to Experments 50 and 62 for related, pertinent data.

PROCEDURE: 1. To 5 ml of each of the four known halogenide solutions in separate test tubes, add 1 ml of calcium nitrate solution. Results? Which ion(s) does this procedure serve to identify?

Observations: . . . . . .

A white precipitate is formed with the fluoride solution. Since no precipitate forms with the other halogenide ions, this test identifies fluoride ion.

2. To 5 ml of each of the four halogenide solutions in separate test tubes, add 1 ml of silver nitrate solution. Results? Write the formula(s) of the precipitate(s) produced, noting the color. Save the precipitate(s) for Part 3. In which case(s) is this procedure not entirely conclusive?

Observations: . . . . . .

No precipitate forms with fluoride. AgCl is white, AgBr is pale yellow, AgI a deeper yellow-tan. The slight difference in colors of the precipitates makes it difficult to reach a conclusion as to whether the unknown is a chloride or a bromide or possibly if it is a bromide or an iodide.

3. To the precipitate(s) formed in Part 2 add 5 ml of ammonia-water and stir. Results? Explain. If solution occurs, add nitric acid until the mixture turns blue litmus paper red. Result? Explain. Which ion(s) will this procedure identify?

Observations: . . . . . .

The AgCl precipitate dissolves completely when stirred with ammonia-water. The AgBr dissolves slightly, the AgI does not dissolve. This solubility in ammonia-water identifies the chloride ion.

4. Separately test 5 ml of chlorine water and 5 ml of potassium chlorate solution according to a scheme which should serve to identify the chloride ion. Results? Explain.

Observations: . . . . . .

Chlorine water gives a positive test yielding a precipitate of AgCl. No precipitate forms with $KClO_3$.

5. To 5 ml of sodium chloride solution add 2 ml of carbon tetrachloride and 1 ml of bromine water. Stopper the test tube and shake it. Permit the test tube to stand. Result? Explain. To 5 ml of potassium bromide solution, add 2 ml of carbon tetrachloride and 1 ml of chlorine water. Shake and permit to settle as before. Results? Explain. To 5 ml of potassium iodide solution, add 2 ml of carbon tetrachloride and 1 ml of chlorine water. Shake and permit to settle. Results? Explain. Repeat the last step using bromine water instead of chlorine water. Results? Explain. What do the results of this series of procedures indicate about the relative strength of the halogens as oxidiz-

ing agents? Which ion(s) may be identified by the use of these procedures?

Observations: The deep orange-red color of Br_2 develops with the bromine water. Bromine does not replace chlorine. Chlorine does replace Br_2 when chlorine water is added to potassium bromide solution yielding the orange-red layer in CCl_4. Both Cl_2 and Br_2 replace iodine in potassium iodide solutions producing the pruple layer in CCl_4. The strength of the halogens as oxidizing agents is: $Cl_2 > Br_2 > I_2$. Bromide and-or iodide ions may be identified by these proceedings.

6. An alternate series of procedures that may be used to identify some of the halogenide ions appears in Experiment 50, Complex Ions of Copper and Silver. Recall Part 2 of this experiment. If you did not perform Experiment 50, perform the series of operations described in Part 2.

7. INSTRUCTOR'S EXPERIMENT. Supplement Part 5, in which potassium iodide, carbon tetrachloride, and chlorine water are used, by continuing to add chlorine water dropwise with shaking until a colorless carbon tetrachloride layer results. This is due to the oxidation of iodine to the iodate ion.

EQUATIONS: Write ionic equations.

1. $2F^- + Ca^{++} \rightarrow CaF_2(s)$

2. Reactions in Part 2.

$Ag^+ + Cl^- \rightarrow AgCl(s)$

$Ag^+ + Br^- \rightarrow AgBr(s)$

$Ag^+ + I^- \rightarrow AgI(s)$

3. Reactions in Part 3. $AgCl + 2NH_3 \rightarrow Ag(NH_3)_2^+ + Cl^-$

Note: a portion of the AgCl may dissolve by forming $Ag(NH_3)_2^+$.

$Ag(NH_3)_2^+ + 2H_3O^+ + Cl^- \rightarrow AgCl(s) + 2H_2O + 2NH_4^+$.

4. Reactions in Part 4.

No reaction

5. Reactions in Part 5.

$2Br^- + Cl_2 \rightarrow 2Cl^- + Br_2$

NAME. . . . . PERIOD . . . . . DATE . . . . . .

INSTRUCTOR'S COMMENTS . . . . . .

Experiment 64 Identification of Salts

PURPOSE: To identify both the positive and the negative ions of unknown salts.

APPARATUS: Beaker, 50 ml; blowpipe and tip; burner and tubing; clamp, buret; cobalt glass; forceps; funnel; medicine dropper; platinum wire rod; ring, iron; ring stand; test tubes.

MATERIAL: Borax, powdered; charcoal or plaster block; iron(II) sulfate; filter paper; ammonia-water solution, dilute (1:4); hydrochloric acid, dilute (1:4); limewater; nitric acid, dilute (1:5); sulfuric acid, concentrated; solutions of: barium chloride (0.1 *M*); cobalt(II) nitrate (0.1 *M*); silver nitrate (0.2 *M*); unknown solids (given out by the instructor).

INTRODUCTION: A chemist may analyze a compound or mixture to find out what it contains, or to learn how much of each element is present. If you have a double laboratory period, you can probably analyze two or three unknowns by using the flame test, the bead test, or the cobalt nitrate test together with the negative ion tests.

It is suggested that you try the flame test first, since it is quicker. If you get a decisive flame coloration and can identify the metallic element present in the unknown given out, it will be unnecessary to try the bead test or the cobalt nitrate test. If no decisive flame coloration is given, then proceed with the cobalt nitrate test. If you do not find this test decisive, then try the bead test.

Next, determine whether the salt given out is a carbonate, a chloride, a sulfate, or a nitrate. An analytical chemist is expected to find all the substances present, and there may be several. As beginners, you will not be given mixtures except where you have learned a procedure for recognizing the components of such mixtures. Always record negative as well as positive results when making an identification test.

PROCEDURE: 1. Test unknown solid *No. 1* by taking up a very small particle on a platinum wire test rod and holding it in the flame as in Experiment 45. Then proceed to make flame tests with unknowns *No. 2* and *No. 3*. If you get a decisive test, record the results in the proper column in the Data Table. If there is no decisive flame coloration, proceed to test a sample of each unknown with the cobalt nitrate test as in Experiment 52. If neither flame test nor cobalt nitrate test gives a decisive result, proceed to make the borax bead test as in Experiment 55.

Observations: Student Answer . . . . . .

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2. To a small portion of each of the unknowns, add a drop of dilute hydrochloric acid. Add additional hydrochloric acid dropwise if necessary to reach a decision. Rapid effervescence of carbon dioxide shows that the salt is a *carbonate*. Sulfites and sulfides also liberate a gas, but in each case the gas has a distinctive odor. Devise a method of identifying the CO_2 gas as it is evolved, using a platinum wire loop and clear limewater solution. Recall the limewater test for CO_2 gas.

Observations: Student Answer . . . . . .

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3. Dissolve a small portion (bulk of a small pea) of each unknown in 15 ml of water in separate test tubes. If a solution is not clear, it should be filtered. Test a 5-ml portion of each solution for chloride ion by first adding to it 5 or 6 drops of dilute nitric acid (why?) and 2 ml of silver nitrate solution. A white

precipitate which is soluble in ammonia-water solution and insoluble in dilute nitric acid shows the presence of a *chloride*.

Observations: Student Answer

4. If the unknowns did not give either a carbonate or a chloride test, then proceed to test them for sulfates as follows: to a 5-ml portion of the solution prepared in Part 3, add 5 or 6 drops of dilute hydrochloric acid (why?) and 2 ml of a solution of barium chloride. A white precipitate shows that a *sulfate* is present.

Observations: Student Answer

5. If one or more of your unknowns did not give a test for a carbonate, chloride, or sulfate, proceed to test it for a nitrate. Dissolve about 0.5 g of green iron(II) sulfate in 10 ml of cold distilled water. Add to it 5 ml of a solution of the unknown as prepared in Part 3. Then add 2 ml of concentrated sulfuric acid, holding the tube in an inclined position so the dense acid will run down the side of the tube without mixing. A brown layer (or ring) at the junction of the acid and the other liquids indicates that a *nitrate* is present.

Observations: Student Answer

(Record in the Data Table both negative and positive results for each test made on each unknown by reporting identifying color, no color, precipitate, no precipitate, odorless gas, or other identifying phrase.)

DATA TABLE

(Student Answers)

Unknown	Flame test	Bead test	$Co(NO_3)_2$ test	CO_3^{--} test	Cl^- test	SO_4^{--} test	NO_3^- test
No. 1							
No. 2							
No. 3							

Unknown Report: Unknown No. 1 .....

Unknown No. 2 .....

Unknown No. 3 .....

NAME . . . . . PERIOD . . . . . DATE . . . . . .

INSTRUCTOR'S COMMENTS . . . . . .

Experiment 65 Radioactivity

PURPOSE: To show how radioactivity may be detected and how the comparative penetrating power of various radiations may be demonstrated. To show that radioisotopes are identical in chemical behavior with isotopes that are not radioactive.

APPARATUS: Simple electroscope; cat's fur; hard-rubber rod; stop watch, or watch with second hand; Geiger counter tube and associated counting apparatus (the Classmaster radioactivity demonstrator is satisfactory); 20 cardboard sheets, 4 inches square, 1/32 inch thick; 15 aluminum sheets, 4 inches square, 1/32 inch thick; 15 lead sheets, 4 inches square, 1/32 inch thick; graph paper.

ADDITIONAL APPARATUS for INSTRUCTOR'S EXPERIMENT: 2 Beakers (100 ml); 2 funnels; glass rods; graduated cylinder; 2 flasks, Erlenmeyer; 2 funnels, separatory; Petri dishes, small; 2 pipets (10 ml) with syringe bulb.

MATERIAL: Radioactive samples: radioactive isotope, if available; radioactive mineral sample, uranium metal or compound, watch with illuminated dial.

ADDITIONAL MATERIAL for INSTRUCTOR'S EXPERIMENT: Carbon tetrachloride; ethyl acetate; iodine-131 (10 microcuries) as KI; uranyl nitrate; filter paper; solutions of: ammonium nitrate (saturated); chlorine water; lead nitrate (0.1 *M*); potassium iodide (0.1 *M*); sodium carbonate (saturated); sodium hydroxide (0.1 *M*).

INTRODUCTION: The types of radiation emitted by radioactive materials include: alpha particles, which are helium nuclei; beta particles, which are electrons; and gamma rays, which are X rays of very short wavelength. Alpha particles will discharge an electroscope but their penetrating power is not great enough to affect a Geiger counter tube. Gamma rays have the greatest penetrating power and may be detected with the Geiger tube shield in place. This metal shield will effectively reflect or absorb most beta particles.

In one part of this experiment, the daughter products of the decay of uranium-238 will be separated from uranyl nitrate. Radioactive iodine-131 (in potassium iodide) will be used in several chemical reactions and its behavior compared with that of iodine-127 which is not radioactive.

SUGGESTIONS: If several counters are available, the class may be organized into teams to perform various, assigned procedures. For example, one team may employ Part 4 with a beta emitter while another team works with a gamma emitter. You may use the beta and gamma disks which are generally part of a demonstration kit or other sources from among those available. Teams may also be assigned to perform parts of the INSTRUCTOR EXPERIMENTS. **CAUTION:** The radioactive sources used in the experiment present no health hazard because the amount of radiation is small. Consult references in the *Teacher Edition* for safety procedures relating to pipeting *(these must never be pipeted orally)*, disposal of radioactive wastes, and care and washing of glassware.

PROCEDURE: 1. *Effect of radioactive materials on an electroscope.* Charge on electroscope by induction using the cat's fur and hard-rubber rod. Observe the rate of discharge of the electroscope by determining how much the leaves collapse during a fifteen-minute period. Now recharge the electroscope and place a radioactive sample beneath the leaves of the electroscope. Observe the rate of discharge of the electroscope during a fifteen-minute period. Other parts of the experiment may be performed in the intervals between these observations.

Observations: The electroscope discharges much more rapidly in the presence of a radioactive sample.

2. *Effect of radioactive materials on a Geiger counter. a. Background count.* Set the Geiger counter apparatus in operation. Note the frequency of clicks when there are no radioactive materials near the counter tube. To what are these counts due? In accurate work this background count must be subtracted from all other measurements.

Observations: The background count is due to ionization caused by cosmic rays or possible radioactive particles on dust.

b. Intensity of radiation. Place the radioactive sample at such a distance from the

DATA TABLE 1

Student answers

Distance	Count	Distance	Count	Distance	Count

DATA TABLE 2

Student answers

Cardboard				Aluminum		Lead	
No. Sheets	Count	No. Sheets	Count	No. Sheets	Count	No. Sheets	Count
0		11		0		0	
1		12		1		1	
2		13		2		2	
3		14		3		3	
4		15		4		4	
5		16		5		5	
6		17		7		7	
7		18		10		10	
8		19		12		12	
9		20		15		15	
10							

Geiger counter tube that you obtain the maximum count reading which the apparatus can measure. Reading Student answers. Remove the radioactive sample, and place the illuminated dial of a wrist watch the same distance from the counter rube. Reading Student answers. If some uranium metal or uranium compound is available, place it the same distance from the counter tube. Reading Student answers. If some radioactive ore or a radioactive isotope is available, test its radioactivity in a similar fashion. Reading Student answers.

3. *Absorptive effect of air on beta particles.* Place a radioactive sample 2 inches from the counter tube. Determine the count. Now place the sample 4 inches from the counter tube. Again determine the count. Continue to move the sample away from the counter tube in 2-inch intervals up to a distance of 24 inches. Make a count reading for each position and record the measurements in Data Table 1. Do not forget to subtract the background count in each case if it is significant.

CURVE: On a piece of graph paper plot a curve of these data, using the distances as abcissas and the counts as ordinates. How far can beta particles travel through the air before being absorbed? Student answer

What type of radiation still reaches the counter tube from the radioactive sample?

Gamma rays

4. *Absorptive effect of other materials on beta radiation.* Place the radioactive sample close to the counter tube so as to obtain the maximum reading of which the counter is capable. Record this count in Data Table 2. Now place one sheet of cardboard between the sample and the counter tube. Make a count reading. Continue to place additional sheets of cardboard between the sample and the counter tube, making a count reading after the addition of each sheet. Use a total of 20 sheets of cardboard.

Repeat the experiment, but use sheets of

aluminum instead of cardboard. Make readings when 1, 2, 3, 4, 5, 7, 10, 12, and 15 sheets of aluminum are placed between the counter and the radioactive sample.

Repeat the experiment, but use sheets of lead this time. Make readings when 1, 2, 3, 4, 5, 7, 10, 12, and 15 sheets of lead are used.

CURVE: Plot curves of these data on the same sheet of coordinate paper, using the number of sheets of various materials as abscissas and the counts as ordinates. Use different colored pencils to draw the graph lines so that the curves may be identified. Which is the most effective material for absorbing beta radiation? Lead is the most effective material for absorbing beta radiation. What must be the relative thickness of cardboard, aluminum, and leads to produce absorption of beta radiation? Student answer indicating that cardboard thickness must be greater than aluminum thickness which must be larger than lead thickness.

5. INSTRUCTOR'S EXPERIMENTS: *a.* Separation of thorium-234 and protactinium-234 from uranium-238. Uranium-238 decays in the sequence:

$$^{238}_{92}\mathrm{U} \rightarrow {}^{4}_{2}\mathrm{He} + {}^{234}_{90}\mathrm{Th}$$

$$^{234}_{90}\mathrm{Th} \rightarrow {}^{0}_{-1}\mathrm{e} + {}^{234}_{91}\mathrm{Pa}$$

$$^{234}_{91}\mathrm{Pa} \rightarrow {}^{0}_{-1}\mathrm{e} + {}^{234}_{92}\mathrm{U}$$

The beta radiations from a sample of uranyl nitrate are being given off by the daughter products. The alpha particles are not detected by the Geiger counter. The procedures used separate the daughter products of uranium-238 by preferential solubility. To $2\bar{0}$ ml of a saturated solution of ammonium nitrate, add 5 grams of uranyl nitrate. Dissolve by stirring. Transfer 15 ml of the resultant solution to the separatory funnel. Test the remaining 5 ml for beta activity, using a small Petri dish as the container. Add 75 ml of ethyl acetate to the separatory funnel and shake for 1 minute. Draw off the lower aqueous layer. Transfer 5 ml of this solution to a small Petri dish. Draw off 5 ml of the ethyl acetate layer to another Petri dish. Test both samples for beta activity. Results?

Observations: The 5 ml of uranyl nitrate dissolved in a saturated solution of ammonium nitrate gives a positive beta activity test. The 5 ml of ethyl acetate solution gives no evidence of beta activity; relatively pure uranyl nitrate is dissolved in the ethyl acetate. The 5 ml of water solution (lower aqueous layer) shows beta activity since the beta-emitting Th and Pa nitrates remain in the water.

b. The chemical behavior of a radioisotope. Iodine-131 is a beta-ray and gamma-ray emitter. Dissolve $1\bar{0}$ microcuries of this isotope in $1\bar{0}$ ml of water. Pipet 5 ml of this solution into a small Petri dish and test it for radioactivity with the Geiger counter. At the same probe distance, test for the different types of radiation by using the tube, first with the shield removed and then with the shield in place. Results? What does this indicate about the relative penetrating power of beta and gamma radiation? With the shiled in place, test for the penetrating power of gamma radiation using Part 4. Record your results in Data Table 3. (p.274)

Observations: Gamma radiation has much greater penetrating power.

6. Place 25 ml of potassium iodide solution in each of two small flasks. Add the 5 ml of the radioactive sample from the Petri dish to one of these. Add $3\bar{0}$ ml of lead nitrate solution to each flask and stir. Add excess lead nitrate solution after the initial precipitate settles to insure complete precipitation. Filter and wash each precipitate (wash bottle). Test each precipitate and 5 ml of each filtrate for radioactivity with the shield covering the Geiger tube. Results? Explain.

Observations: The precipitate is radioactive, the filtrate is not radioactive.

A further experiment may be tried by the reaction of each precipitaet with $1\bar{0}$ ml of saturated sodium carbonate solution. The yellow lead iodide precipitate is converted to white lead carbonate which is removed by filtration. After washing, these precipitates as well as the initial filtrates are tested for gamma radiation.

Observations: The lead carbonate precipitate is radioactive, the filtrate is not radioactive.

7. Dilute 1 ml of the remaining radioactive solution to 25 ml. Add 3 drops of sodium hydroxide solution. Add 2 crystals of nonradioactive potassium iodide and stir into solution. Measure the gamma activity of 5 ml of this solution in a Petri dish. Pour 15 ml of this solution into a separatory funnel. Add 10 ml of carbon tetrachloride and 3 drops of chlorine water. Shake the separatory funnel and permit the carbon tetrachloride layer to settle. Result? Explain. Separate this layer from the water layer. Test 5 ml samples of each layer in small Petri dishes with the Geiger counter. Results? Explain. What do the results of these

DATA TABLE 3

Student answers

Cardboard				Aluminum		Lead	
No. Sheets	Count	No. Sheets	Count	No. Sheets	Count	No. Sheets	Count
0		11		0		0	
1		12		1		1	
2		13		2		2	
3		14		3		3	
4		15		4		4	
5		16		5		5	
6		17		7		7	
7		18		10		10	
8		19		12		12	
9		20		15		15	
10							

procedures show about the chemical properties of radioisotopes?

Observations: Violet CCl_4 solution is due to oxidation of I^- to I_2. The CCl_4 layer is radioactive. The results of this experiment and parts 5b and 6 indicate that radioisotopes have the same chemical properties as the isotopes that are not radioactive.

FURTHER EXPERIMENTATION: The Union Carbide MINIGENERATOR and the corresponding instruction manual may be purchased from W. H. Curtin Scientific Co., P.O. Box 1546, Houston, Texas 77001 or from the Welch Scientific Supply Co., 7200 N. Linder Avenue, Skokie, Ill. 60076. It is available in two systems: Cesium-137 yields Barium-137m (2.6 minutes half-life) and Tin-113 yields Indium-113m (100 minutes half-life). Among the twenty five experiments is one on the half-life of these short-lived isotopes, which are readily available for as many as 1000 repeated uses by use of the MINIGENERATOR.

Appendix

List of Apparatus for Each Student Locker

Asbestos square (6″ × 6″ × 1/16″)
Beakers, 1 (50 ml); 1 (150 ml); 1 (250 ml); 1 (400 ml).
Blowpipe and tip.
Bottles, wide mouth, 4 (6 oz).
Brush, test tube.
Burner, Bunsen or Tirrell, and 2 ft tubing.
Clamp, buret.
Clamp, test tube.
Crucible and cover (size 1, low form).
Dishes, evaporating, 2 (size 0).
File, triangular.
Filter paper, 1 pkg. (12.5 cm).
Flasks, Erlenmeyer, 2 (125 ml).
Flask, Florence (250 ml).
Forceps (5″).
Funnel (65 mm, short stem).
Funnel tube or thistle tube.
Glass plates, 4 (3″ × 3″ × 1/8″).
Graduated cylinder, 1 (10 ml); 1 (50 ml).
Magnifying lens.
Medicine dropper.
Metric ruler (plastic, 15 cm).
Mortar and pestle (size 00).
Rubber connectors, 2 (thin wall 3/16″ inside diam.).
Spatula, spoon at one end (6″).
Splints, wooden, bundle of 50.
Spoon, deflagrating.
Test tubes, Pyrex, 10 (18 mm × 150 mm); 2 (25 mm × 200 mm).
Test tube rack.
Tongs.
Triangle, pipestem (2″ on a side).
Tubing, delivery, rubber, 2 ft (medium wall, 3/16″ inside diam.).
Watch glasses, 2 (3″).
Wing top, or flame spreader.
Wire gauze, asbestos center (5″ × 5″).

Additional Laboratory Equipment (General)

Apparatus for temporary loan, occassional use, or demonstration. Quantity required for a class of 30 pupils, not including excess to cover breakage.

Acid pitcher (2 qt).
Adapter, bent, glass.
Asbestos sheet, 1/8 in(3).
Balance, analytical, and set of precision weights.
Balances, 15 (triple beam, sensitive to decigrams); or 15 (Harvard Trip Balances with sets of weights).
Balances, 15 (triple beam, sensitive to centigrams)
Barometer.
Bath, sand (5″, shallow).
Bath, water, 30 (6″ diam.).
Battery jars, 15 (6″ diam. × 8″ high).
Beakers, 24 (400 ml); 24 (600 ml); 12 (1000 ml).
Bottle, 1 (1 gal.).
Bottles, dropping, 60 (30 ml).
Bottles, plastic capped or glass stoppered, narrow mouth, 60 (250 ml).
Bottles, reagent, 100 (250 ml), 100 (500 ml).
Bottles, reagent, salt mouth, plastic capped, 1 gross, (125 ml); 1/2 gross, (250 ml); 1/4 gross, (500 ml).
Brushes, beaker, 6: counter, 6 funnel, 6.
Burets, 30 (50 ml for bases); 30 (50 ml for acids).
Burners, large, Meker or Fisher, 3.
Clamps, pinch, 60.
Clamps, condenser, 30.
Clamps, double, for burets, 30.
Cobalt glass plates. 60 (2″ × 2″ × 1/8″).
Condenser, Liebig, 30 (20″ long).
Conductivity apparatus, (6).
Cork borers, set.
Corks, assorted sizes 0-11, 1 gross.
Corks, 60 (size 8).
Crucible, sand, 1.
Cylinders, graduated, 6 (100 ml); 15 (250 ml); 3 (500 ml); 3 (1000 ml).
Demonstration cells, with porous cup, 15.
Diffraction grating, card-mounted (15).
Dishes, evaporating, large, 2.
Dry cells, 45.
Drying oven, small.
Electrodes, 15 carbon; 15 copper; 15 platinum; 15 zinc.
Electrolysis apparatus, demonstration.
Electroscopes, 15.
Eudiometers, 30 (50 ml).
Filter paper, large, 1 pkg.
Flasks, distilling, 30 (250 ml); 1 (500 ml).
Flasks, Erlenmeyer, 3 (250 ml); 6 (500 ml); 6 (1000 ml).
Flasks, Florence, 30 (500 ml).
Flasks, volumetric, 2 (1000 ml); 1 (500 ml); 30 (250 ml); 1 (100 ml).
Friction pad, rubber, 15.
Friction rods, hard rubber, 15.
Funnels, 6 (4″ diam.); 6 (8″–10″ diam.)
Funnels, separatory, stoppered, 30 (125 ml).
Hammer, claw.
Labels, gummed, 12 boxes.
Lead dishes, 30 (250 ml).
Magnets, bar, 15 pairs.
Meter sticks, (15) one meter; (15) half-meter.

Meters
Meter stick supports, low form (60).
Meter stick lens support (15)
Mortars and pestles, 1 (porcelain, 6″ diam.); 1 (iron, 6″ diam.).
Nuclear demonstrator, with extendable probe.
Paper rolls, with rack, 2 (6″ wide); extra rolls of paper, 4.
Pans, enameled, rectangular, 5 (9″ × 6″ × 2″).
Pipets, 30 (10 ml).
Pipet, bulb, 1.
Pipet, graduated, 1 (10 ml).
*Plastic cups and covers, (12 oz), 60 as calorimeters.
*Thermo Kups, Mid-West-Pak, Belvidere, Ill., obtainable from Lebanon Valley Paper Co., 105 N. 16th St., Lebanon, Pa.
Platinum wire, No. 24, 5 ft.
Pliers and wire cutter.
Printing frames, 15.
Radioactive sources.
Retorts, glass stoppered, Pyrex, 30 (250 ml).
Ring stands, with 3 rings each, 60.
Rod, glass, 2 lb (6 mm diam.).
Scissors, 1 pr. (6″).
Screwdriver.
Shears, heavy, for cutting metals, 1 pr. (12″).
Shield, metal, for distilling flask.
Shields, wire-mesh reinforced glass, 2.
Spatula, porcelain (25 cm).
Stoppers, rubber, assorted sizes 0-6, solid, 1-hole, and 2-hole, 6 lb.
Suction (outlet) pump, or aspirators.
Thermometers, 30 (−20° C to 110° C); 3 (−10° C to 360° C).
Tripods, with concentric rings, 6 (8″).
Troughs, pneumatic, 30.
*Tubes, Charles' Law Tube, mounted, 30; drying, calcium chloride, 30; volume coefficient tube. 40 cm capillary, 30.
Tubing, glass, 5 lb (5 mm); 10 lb (6 mm).
Tubing, rubber, thin wall, 3/16″ inside diameter, 10 ft.
Tubing, rubber, medium wall, 3/16″ inside diameter, 60 ft.
Tubing, rubber, medium wall, 1/4″ inside diameter, 180 ft.
Vials, shell, 30 (2 dram capacity).
Voltmeters, D. C., 0-1.5 v, 15.
Water still, automatic.
Wire gauze, bright, plain, 30.

List of chemicals needed for 30 pupils

Acetamide, practical. 500 g.
Acetic acid, glacial, reagent. 3 lb.
Acetone, reagent. 1 pt.
Alka-Seltzer, tablets. 2 packages.
Albumin, from eggs, soluble, powder. ¼ lb.
Aluminum, metal, granular, reagent. 1 lb; sheet, 1/32″, 1 lb.
Aluminum chloride, reagent. 1 lb.
Aluminum sulfate, reagent. 1 lb.
Aluminum sulfide, pure. 4 oz.
Ammonium acetate, reagent. 1 lb.
Ammonium carbonate, powder, C.P. 1 lb.
Ammonium chloride, granular, U.S.P. 1 lb.
Ammonium hydroxide, reagent. 5 pt.
Ammonium nitrate, reagent. 1 lb.
Ammonium sulfate, granular, white, purified. 1 lb.
Ammonium sulfide, light, reagent. 1 pt.
Ammonium thiocyanate, crystal, reagent. ¼ lb.
Amyl alcohol, iso-pentyl alcohol. 1 pt.
Aniline hydrochloride, pure. 1 lb.
Antimony, metal, powder. ¼ lb.
Antimony trichloride, crystal, reagent. ¼ lb.
Asbestos, fiber. ½ lb.
Asbestos paper. 1 roll.
Baking powder. 2 cans.
Barium chloride, crystal, reagent. 1 lb.
Barium hydroxide, crystal, reagent. ¼ lb.
Barium nitrate, crystal, reagent. ¼ lb.
Barium peroxide, C.P., 4 oz.
Barium sulfate, C.P. ¼ lb.
Beef suet. 1 oz.
Benzene, Pure. 1 qt.
Benzoic acid, Pure. 4 oz.
Boric acid, reagent. 1 lb.
Bromine, N.F. ¼ lb.
Bromine water. 1 lb.
Bromthymol blue, reagent. 1 oz.
Butyric acid, 98-100%, technical. 1 pt.
Cadmium nitrate, reagent. ¼ lb.
Cadmium nitrate, reagent. ¼ lb.
Calcium carbide, granular. 1 lb.
Calcium carbonate, powder, pure. 1 lb.
Calcium carbonate (Marble chips). 5 lb.
Calcium chloride, anhydrous, granular, 4-8 mesh. 5 lb.
Calcium fluoride, powder, reagent. ¼ lb.
Calcium hydroxide, U.S.P. 1 lb.
Calcium hypochlorite (or bleaching powder) 1 lb.
Calcium metal. ¼ lb.
Calcium nitrate, crystal, reagent. 1 lb.
Calcium oxide, lump, N.F. 1 lb.
Calcium sulfate, dry, powder, reagent, ¼ lb.
Camphor, synthetic block, U.S.P. 1 lb.
Candles, paraffin, 5 in. 1 doz.
Carbon disulfide, purified. 5 lb.
Carbon tetrachloride, technical. 2 qt.
Carnation, red. 30.
Charcoal, activated. 2 lb.
Charcoal, animal (Boneblack), powder, technical, 1 lb.
Charcoal, wood, lump. 1 lb.

Charcoal wood, powder, technical. 1 lb.
Charcoal, wood, sticks, for blowpipe work. 3 doz.
Chlorine water. 1 lb.
Chromium (III) sulfate, granular, reagent. ¼ lb.
Clorox.
Cobalt(II) chloride, crystal, reagent. ¼ lb.
Cobalt(II) nitrate, crystal, reagent. ¼ lb.
Copper(II) carbonate, technical. 1 lb.
Copper, metal, foil, 1/100" thick. 1 lb.
Copper, metal turnings. 2 lb.
Copper, wire, bare, No. 16. 1 lb.
Copper, wire, bare, No. 18. ¼ lb.
Copper, wire, bare, No. 22 or No. 26. ¼ lb.
Copper, wire, insulated, No. 18. ½ lb.
Copper(II) carbonate, technical, 1 lb.
Copper(II) nitrate, crystal, reagent. ¼ lb.
Copper(II) oxide, black, powder, technical. 1 lb.
Copper(II) oxide, black, wire, reagent. ¼ lb.
Copper(II) sulfate, anhydrous, reagent. ¼ lb.
Copper(II) sulfate, granular, U.S.P. 3 lb.
Copper(II) sulfate crystals, technical ($CuSO_4 \cdot 5H_2O$). 5 lb.
Cotton, absorbent. 1 lb.
Cotton cloth, colored, 1 yd.
Cottonseed oil, U.S.P. 1 pt.
Detergent, granular,
Diarsenic trioxide, powder, N.F. 1 lb.
Ethyl acetate, C.P. 1 pt.
Ethyl alcohol (Ethanol), 95%, U.S.P. 1 pt.
Ethyl alcohol (Ethanol), denatured. 1 gal.
Ethylene glycol. 1 pt.
Fehling's solution A. 1 lb.
Fehling's solution B. 1 lb.
Formaldehyde, pure. 1 lb.
Formic acid, 85-90%, reagent. ½ pt.
Gasoline, 1 pt each of several grades.
Gelatin, granular, U.S.P. 1 lb.
Glycerol (Glycerin), white, U.S.P. 1 pt.
Graphite, powder. 1 lb.
Hydrion test paper, wide range, type A. 5 vials.
Hydrion test paper, wide range, type b. 5 vials.
Hydrion test paper, type D. 5 vials.
Hydrochloric acid, concentrated, reagent. 24 lb.
Hydrogen peroxide, 30%, C.P. 1 pt.
Hydrogen peroxide, 3%, U.S.P. 1 pt.
Ink, India. 1 bottle.
Iodine, resublimed, U.S.P. ¼ lb.
Iron, metal, filings, fine. 1 lb.
Iron, metal, wire, cut (Card teeth) N.F. 1 lb.
Iron, nails, 3". 2 lb.
Iron(II) ammonium sulfate, granular, reagent. ¼ lb.
Iron(II) (III) oxide (magnetite), powdered. 1 lb.
Iron(II) sulfate, crystal, U.S.P. 1 lb.
Iron(II) sulfide, broken lumps. 2 lb.
Iron(III) ammonium citrate, green, U.S.P. 1 lb.
Iron(III) chloride, lump, U.S.P. 1 lb.
Iron(III) nitrate, crystal, reagent. ¼ lb.
iso-Amyl alcohol, 85%. 1 pt.
Kerosene. 1 qt.
Lampblack, 1 lb.
Lead, metal, foil, 1/1000" thick. 1 lb.
Lead, metal, sheet, 1/32" thick. 1 lb.
Lead(II) acetate, granular, N.F. 1 lb.
Lead(II) nitrate, crystal, reagent. ¼ lb.
Lead (II, IV) oxide, pure. 1 lb.
Lead (IV) oxide, pure. 1 lb.
Lead sulfide, powdered, technical. 1 lb.
Lithium nitrate, reagent. ¼ lb.
Litmus paper, blue. 30 vials.
Litmus paper, red. 30 vials.
Litmus, powder. ¼ lb.
Magnesium, metal, ribbon. 2 oz.
Magnesium, metal, turnings. 4 oz.
Magnesium chloride, crystal, N.F. 1 lb.
Magnesius hydroxide, "milk of magnesia." 1 bottle.
Magnesium sulfate, crystal, U.S.P. 1 lb.
Manganese dioxide, powder, reagent. 1 lb.
Manganese dioxide, granular, technical. 1 lb.
Manganese nitrate, crystal, C.P. ½ lb.
Manganese(II) sulfate, reagent. ½ lb.
Mercury(I) nitrate, crystal, reagent. 1 oz.
Mercury(II) chloride, granular, U.S.P. ¼ lb.
Mercury(II) nitrate, crystal, reagent. 1 oz.
Methyl alcohol (Methanol), absolute, synthetic, purified. 1 pt.
Methylene blue. 1 oz.
Methyl orange (indicator), reagent. 1 oz.
Mineral oil.
Molasses. 1 pt.
Nickel(II) chloride, reagent. ¼ lb.
Nickel(II) sulfate, crystal, reagent. ¼ lb.
Nitric acid, concentrated, reagent. 28 lb.
Oil, olive, pure. 1 pt.
Oxalic acid, pure. 1 lb.
Phenolphthalein, powder, U.S.P. ¼ lb.
Phosphoric acid, reagent. 1 pt.
Phosphorus, red, amorphous. ¼ lb.
Phosphorus, white (yellow), thick sticks, N.F. ¼ lb.
Potassium, metal. 1 oz.
Potassium bromide, granular, U.S.P. 1 lb.
Potassium carbonate, granular. N.F. 2 lb.
Potassium chlorate, small crystal, reagent. 2 lb.
Potassium chloride, white, granular, U.S.P. 2 lb.
Potassium chromate, reagent. ¼ lb.
Potassium cyanide, reagent. ¼ lb.
Potassium dichromate, technical. 1 lb.
Potassium hexacyanoferrate(II), crystal, reagent. ¼ lb.
Potassium hexacyanoferrate(III), crystal, reagent. ¼ lb.

Potassium hydrogen carbonate (bicarbonate), crystal U.S.P. 1 lb.
Potassium hydrogen tartrate (bitartrate), powder, N.F. 1 lb.
Potassium hydroxide, pellets, reagent. ¼ lb.
Potassium iodide, crystal, U.S.P. ½ lb.
Potassium nitrate, crystal, reagent. 2 lb.
Potassium permanganate, crystal, U.S.P. 1 lb.
Potassium sulfate, crystal, reagent. 1 lb.
Potassium thiocyanate, reagent. ¼ lb.
Pyrogallol (Pyrogallic acid), crystal, reagent, 1 oz.
Salicylic acid, crystal, reagent. ¼ lb.
Sand. 5 lb.
Sandpaper, fine. 4 sheets.
Silver, metal, foil 1/100" thick. 3 oz.
Silver acetate, purified. ½ lb.
Silver nitrate, crystal, reagent. ½ lb.
Soap, castile, white, powder, N.F. 1 lb.
Soda lime, 8-14 mesh. 1 lb.
Sodium, metal. ¼ lb.
Sodium acetate, fused, anhydrous, reagent. ¾ lb.
Sodium acetate, crystal, reagent. ¼ lb.
Sodium aluminum sulfate, technical. ½ lb.
Sodium borate, tetra-, powder, U.S.P. 1 lb.
Sodium carbonate, anhydrous, reagent. ¼ lb.
Sodium carbonate, crystal, reagent. 1 lb.
Sodium chloride, crystal, reagent. ½ lb.
Sodium chloride, white, technical (Table salt). 5 lb.
Sodium fluoride, technical. 1 lb.
Sodium hydrogen carbonate, powder, U.S.P. 1 lb.
Sodium hydrogen sulfate, reagent. 1 lb.
Sodium hydroxide, pellets, reagent. 1 lb.
Sodium hydroxide, flakes, reagent. 1 lb.
Sodium hypochlorite (Chlorox). 1 gal.
Sodium nitrate, crystal, reagent. 2 lb.
Sodium nitrite, granular, U.S.P. 1 lb.
Sodium oxalate, reagent. ¼ lb.
Sodium peroxide, reagent, 20 mesh and finer. ¾ lb.
Sodium phosphate, tribasic, reagent. ¼ lb.
Sodium sulfate, small crystal, U.S.P. 2 lb.
Sodium sulfide, crystal, reagent. ¼ lb.
Sodium sulfite, dry, powder, N.F. 1 lb.
Sodium thiosulfate, crystal, reagent. ½ lb.
Starch, corn. 1 lb.
Strontium chloride, reagent. ¼ lb.
Strontium nitrate, reagent. ¼ lb.
Sucrose, granular (Granulated sugar). ½ lb.
Sugar, dark brown. 1 lb.
Sulfur, sublimed (Sulfur flowers). 2 lb.
Sulfur, roll, technical. 2 lb.
Sulfuric acid, concentrated, reagent. 36 lb.
Taconite, powdered ore. 1 lb.
Tartaric acid, powder, reagent. ¼ lb.
Thermite, black. 1 lb.
Thermite igniting mixture, ½ lb.
Thread, cotton, No. 40. 1 spool.
Tin, metal, mossy, purified. ¼ lb.
Tin(II) chloride, crystal, reagent. ¼ lb.
Uranyl nitrate, C.P. 1 oz.
Uranium ore. 1 oz.
Vinegar. 2 pt.
Yeast. 1 package.
Zeolite water softener. 2 lb.
Zinc, metal, mossy, technical, 2 lb.
Zinc, metal powder (dust), technical. 1 lb.
Zinc, metal, sheet, 1/100" thick. 1 lb.
Zinc chloride, dry, granular, N.F. 1 lb.
Zinc nitrate, crystal, reagent. ¼ lb.
Zinc sulfate, granular, U.S.P. 1 lb.
Zinc sulfide ore, powdered, 1 lb.

Purchasing Suggestions

Sulfuric acid is commonly sold in plastic capped bottles holding 9 lb of the acid; nitric acid in bottles holding 7 lb; hydrochloric acid in bottles holding 6 lb; and ammonia-water solution (ammonium hydroxide) in bottles holding 4 lb. Each of these is packed in six disposable bottles in a disposable cardboard carton. It will be advisable to order a carton of each of these, even though the supply lasts more than one year, because of lower shipping costs and a more favorable price.

While commercial grade acid can be used for some purposes, reagent grade acid is needed for many purposes. It is recommended that only reagent grade sulfuric, nitric, and hydrochloric acids be ordered. The cost per student is only a little more, and the results are much better.

Purchase only reagent grade manganese dioxide. Organic impurities in manganese dioxide have been known to cause an explosion in the oxygen experiment.

Calcium oxide deteriorates so rapidly that it is advisable to buy only a small quantity fresh each year. Likewise, hydrogen peroxide must be purchased fresh each year.

Hydrion dual-range sets of various types may be used instead of the vial loose papers.

Preparation of Solutions

In making up solutions, distilled water should be used. The amount of solution to be made up will vary with the experiment in which it is used and the size of the class or classes. This is also true of the number and size of the bottles used for dispensing the solution. Consequently no general suggestions can be given.

For convenience, this list has been prepared with the amounts of materials needed for making up one liter of each solution. The instructor can readily make his own adjustments when preparing smaller or larger quantities.

Unless otherwise stated, dissolve the amount of the material indicated in distilled water and dilute to one liter.

Acetic acid: **6 M**, 344 ml of glacial acetic acid; **30%**, 300 ml of glacial acetic acid; **4 M** (app. N), **Dilute** (1:3), 250 ml of glacial acetic acid; **Dilute** (1:4), 200 ml of glacial acetic acid; **0.1 M**, 6 ml of glacial acetic acid.

Aluminum chloride: **0.2 M**, 27 g of $AlCl_3$.

Aluminum sulfate: **0.1 M**, 67 g of $Al_2(SO_4)_3 \cdot 18\ H_2O$; 0.03 M, 20 g of $Al_2(SO_4)_3 \cdot 18\ H_2O$; **0.02 M**, 13 g of $Al_2(SO_4)_3 \cdot 18\ H_2O$.

Ammonia-water solution: **6 M**, 400 ml of concentrated ammonia-water solution; **4 M**, 270 ml of concentrated ammonia-water; **Dilute** (1:4), 200 ml of concentrated ammonia-water solution; **1 M**, 70 ml of concentrated ammonia-water solution; **0.1 M**, 7 ml of concentrated ammonia-water solution.

Ammonium acetate: **0.1 M**, 8 g of $NH_4C_2H_3O_2$.

Ammonium chloride: **0.1 M**, 5 g of NH_4Cl.

Ammonium sulfide: **0.3 M**, 120 g of ammonium sulfide solution assaying 8% sulfidic sulfur. Use a brown bottle.

Aniline hydrochloride, 180 g.

Antimony trichloride: **0:25 M**, Dissolve 56 g of $SbCl_3$ in water to which 25 ml of conc. hydrochloric acid has been added. Dilute to one liter.

Barium chloride: **0.5 M**, 122 g of $BaCl_2 \cdot 2\ H_2O$; **0.25 M**, 61 g of $BaCl_2 \cdot 2\ H_2O$; **0.1 M**, 24 g of $BaCl_2 \cdot 2\ H_2O$.

Barium hydroxide: **0.2 M**, 63 g of $Ba(OH)_2 \cdot 8\ H_2O$.

Barium nitrate: **0.5 M**, 131 g of $Ba(NO_3)_2$.

Bromine water: Dissolve 1 ml of liquid bromine in water and dilute to one liter. Use a brown bottle.

Bromthymol blue: **0.04%**, 0.1 g by mixing dry in a mortar with 16.0 ml of 0.01 M NaOH and dilute to 250 ml with water.

Cadmium nitrate: **0.25 M**, 77 g of $Cd(NO_3)_2 \cdot 4\ H_2O$.

Calcium chloride: **0.5 M**, 110 g of $CaCl_2 \cdot 6\ H_2O$.

Calcium nitrate: **0.5 M**, 82 g of $Ca(NO_3)_2$.

Calcium sulfate: **Sat'd**, Add one liter of water to 5 g of $CaSO_4 \cdot 2\ H_2O$.

Chlorine water: Saturate one liter of water with chlorine gas. Use a brown bottle.

Chromium sulfate: **0.1 M**, Dissolve 72 g of $Cr_2(SO_4)_3 \cdot 18\ H_2O$ in one liter.

Cleaning solution: Add 400 ml of concentrated sulfuric acid to 50 g of potassium dichromate $K_2Cr_2O_7$. This fluid may be used to clean laboratory glassware, such as beakers, test tubes, and burets by letting the solution stand in them overnight. The solution may be used again and again. It should not be thrown away after use.

Cobalt(II) choride: **0.1 M**, 24 g of $CoCl_2 \cdot 6\ H_2O$.

Cobalt(II) nitrate: **0.1 M**, 29 g of $Co(NO_3)_2 \cdot 6\ H_2O$; **0.17 M**, 49 g of $Co(NO_3)_2 \cdot 6\ H_2O$.

Copper(II) nitrate: **0.25 M**, 74 g of $Cu(NO_3)_2 \cdot 6\ H_2O$; **0.2 M**, 59 g of $Cu(NO_3)_2 \cdot 6\ H_2O$; **0.1 M**, 30 g of $Cu(NO_3)_2 \cdot 6\ H_2O$.

Copper(II) sulfate: **1 M**, Dissolve 250 g of $CuSO_4 \cdot 5\ H_2O$ in water to which 5 ml of conc. sulfuric acid has been added. Dilute to one liter; **0.25 M**, Dissolve 63 g of $CuSO_4 \cdot 5H_2O$ in water to which 5 ml of conc. sulfuric acid has been added. Dilute to one liter; **0.2 M**, Dissolve 50 g of $CuSO_4 \cdot 5\ H_2O$ in water to which 5 ml of conc. sulfuric acid has been added. Dilute to one liter; **0.1 M**, Dissolve 25 g of $CuSO_4 \cdot 5\ H_2O$ in water to which 5 ml of conc. sulfuric acid has been added. Dilute to 1 liter; **0.05 M**, 13 g of $CuSO_4 \cdot 5H_2O$.

Formalin: 40% formaldehyde, 400 g of formaldehyde in 600 ml of water.

Glycerol: **6 M**, 552 g of $C_3H_5(OH)_3$.

Hydrochloric acid: **6 M**, 516 ml of conc. hydrochloric acid; **Dilute (1:3)**, 250 ml of conc. hydrochloric acid; **Dilute (1:4)**, 200 ml of conc. hydrochloric acid; **0.1 M**, 9 ml of conc. hydrochloric acid; **0.1 N** standard solution, 8.6 ml of conc. hydrochloric acid.

Hydrogen peroxide: 6%, 200 g of 30% hydrogen peroxide, H_2O_2 in 800 ml of water.

Hydrogen sulfide: **Sat'd**, Saturate one liter of water with hydrogen sulfide gas; **Very dilute**, Use 25 ml of a saturated solution of hydrogen sulfide in water and dilute to one liter with water.

Iodine: 3 g of KI and 0.6 g of I_2 in 200 ml of water.

Iron(II) ammonium sulfate: **0.1 M**, Dissolve 39 g of $Fe(NH_4)_2(SO_4)_2 \cdot 6\ H_2O$ in water to which 10 ml of conc. Sulfuric acid has been added. Dilute to one liter. Should be freshly prepared.

Iron(II) sulfate: **0.25 M**, Dissolve 70 g of $FeSO_4 \cdot 7\ H_2O$ in water to which 10 ml of conc. sulfuric acid has been added. Dilute to one liter. Add 6 small iron nails to the solution. Should be freshly prepared. **0.1 M**. Dissolve 28 g of $FeSO_4 \cdot 7\ H_2O$ in water to which 10 ml of conc. sulfuric acid has been added. Dilute to one liter. Add 6 small iron nails to the solution. Should be freshly prepared.

Iron(II) chloride: React steel wool with 6 M HCl permitting excess of steel wool to keep in Fe(II) state.

Iron(III) chloride: **1 M**, Dissolve 270 g of $FeCl_3 \cdot 6\ H_2O$ in water to which 20 ml of conc. hydrochloric acid has been added. Dilute to one liter; **0.1 M**, Dissolve 27 g of $FeCl_3 \cdot 6\ H_2O$ in water to which 20 ml of conc. hydrochloric acid has been added. Dilute to one liter; **0.07 M**, Dissolve 19 g of $FeCl_3 \cdot 6\ H_2O$ in water to which 20 ml of conc. hydrochloric acid has been added. Dilute to one liter; **0.04 M**, 11 g of $FeCl_3 \cdot 6\ H_2O$. Dilute to one liter.

Lead(II) acetate: **0.25 M**, 95 g of $Pb(C_2H_3O_2) \cdot 3\ H_2O$.

Lead(II) nitrate: **0.1 M**, 33 g of $Pb(NO_3)_2$.

Limewater: Add one liter of water to 5 g of $Ca(OH)_2$. Shake well and allow the undissolved solids to settle. Decant the saturated solution to another bottle.

Lithium nitrate: **0.5 M**, 35 g of $LiNO_3$.

Litmus: Add 250 ml of boiling water to 25 g of litmus powder. Allow to stand 15 minutes. Decant the liquid. Add 500 ml of boiling water. Let the solution stand overnight. Filter.

Magnesium chloride: **0.1 M**, 20 g of $MgCl_2 \cdot 6\ H_2O$.

Magnesium hydroxide: **saturated**, dilute commercial "milk of magnesia" and filter.

Magnesium sulfate: **0.5 M**, 123 g of $MgSO_4 \cdot 7\ H_2O$; 200 **p.p.m.**, 0.2 g $MgSO_4 \cdot 7\ H_2O$.

Manganese(II) sulfate: **0.25 M**, 69 g of $MnSO_4 \cdot 7\ H_2O$.

Mercury(I) nitrate: **0.1 M**, Dissolve 56 g of $HgNO_3 \cdot H_2O$ in water to which 50 ml of conc. nitric acid has been added. Dilute to one liter.

Mercury(II) chloride: **0.1 M**, Dissolve 27 g of $HgCl_2$ in water to which 20 ml of conc. hydrochloric acid has been added. Dilute to one liter.

Mercury(II) nitrate: **0.1 M**, Dissolve 32 g of $Hg(NO_3)_2$ in water to which 20 ml of conc. nitric acid has been added. Dilute to one liter.

Methyl orange: 1 g of methyl orange.

Nitric acid: **Dilute** (1:4), 200 ml of concentrated nitric acid; **Dilute (1:5)**, 165 ml of concentrated nitric acid; **0.1 M**, 7 ml of concentrated nitric acid.

Oxalic acid: **Sat'd**,100 g of $(COOH)_2 \cdot 2\ H_2O$, 0.1 M (8.2 g).

Phenolphthalein: Dissolve 10 g of phenolphthalein in 500 ml of denatured alcohol and add 500 ml of water.

Phosphoric acid: **Dilute** (1:14), 67 ml of concentrated phosphoric acid.

Potassium bromide: **0.5 M**, 60 g of KBr: **0.2 M**, 24 g of KBr.

Potassium carbonate: **0.05 M**, 7 g of K_2CO_3.

Potassium chloride: **0.2 M**, 15 g of KCl; **0.1 M**, 7 g of KCl.

Potassium chromate: **0.1 M**, 20 g of K_2CrO_4; **0.25 M**, 50 g of $K_2Cr_2O_4$.

Potassium cyanide: **0.2 M**, 13 g of KCN.

Potassium hexacyanoferrate(II): **0.1 M**, 42 g of $K_4Fe(CN)_6 \cdot 3\ H_2O$.

Potassium hexocyanoferrate(III): **Sat'd**, 350 g of $K_3Fe(CN)_6$; **0.1 M**, 33 g of $K_3Fe(CN)_6$.

Potassium hydrogen tartrate: **0.5 M**, 94 g of $KHC_4H_4O_6$.

Potassium hydroxide: **2.5 M**, 150 g of KOH; **1 M**, 56 g of KOH.

Potassium iodide: **0.5 M**, 83 g of KI; **0.2 M**, 33 g of KI; **0.1 M**, 16 g of KI.

Potassium nitrate: **0.5 M**, 51 g of KNO_3; **0.1 M**, 10 g of KNO_3.

Potassium permanganate: **0.1 M**, 16 g of $KMnO_4$; **0.02 M**, 3 g of $KMnO_4$.

Potassium thiocyanate: **1 M**, 97 g of KCNS; **0.2 M**, 19 g of KCNS.

Silver acetate: **Sat'd**, 11 g of $AgC_2H_3O_2$.

Silver nitrate: **0.2 M**, 34 g of $AgNO_3$. Use a brown bottle. **0.1 M**, 17 g of $AgNO_3$. Use a brown bottle.

Soap: 2%, Dissolve 20 g of dry castile soap in one liter of 80% alcohol (5 parts alcohol to 1 part water).

Sodium acetate: **0.1 M**, 8 g of fused $NaC_2H_3O_2$.

Sodium aluminum sulfate: **0.25 M**, 115 g of $NaAl(SO_4)_2 \cdot 12\ H_2O$.

Sodium carbonate: **0.3 M**, 32 g of Na_2CO_3; 2%, 20 g of Na_2CO_3; **0.05 M**, 5 g of Na_2CO_3.

Sodium chloride: **0.5 M**, 29 g of NaCl; **0.2 M**, 12 g of NaCl; **0.1 M**, 6 g of NaCl.

Sodium fluoride: **0.1 M**, 4 g of NaF.

Sodium hydrogen carbonate: **0.1 M**, 8 g of $NaHCO_3$.

Sodium hydroxide: **10 M**, 400 g of NaOH; **6 M**, 240 g of NaOH; **3 M**, 120 g of NaOH; **2.5 M**, 100 g of NaOH; **2 M**, 80 g of NaOH; **1 M**, 40 g of NaOH; **0.5 M**, 20 g of NaOH; **0.5 M**, 20 g of NaOH; **0.1 M**, 4 g of NaOH.

Sodium nitrate: **0.5 M**, 43 g of $NaNO_3$.

Sodium oxalate: **0.1 M**, 7 g of $Na_2C_2O_4$.

Sodium phosphate: **0.04 M**, 15 g of $Na_3PO_4 \cdot 12\ H_2O$.

Sodium sulfate: **1 M**, 142 g of Na_2SO_4; **0.1 M**, 14 g of Na_2SO_4; **0.05 M**, 7 g of Na_2SO_4.

Sodium sulfide: **0.1 M**, 24 g of $Na_2S \cdot 9\ H_2O$.

Sodium tetraborate: **2%**, 20 g of $Na_2B_4O_7 \cdot 10\ H_2O$.

Sodium thiosulfate: **2 M**, 496 g of $Na_2S_2O_3 \cdot 5\ H_2O$; **0.25 M**, 62 g of $Na_2S_2O_3 \cdot 5\ H_2O$.

Starch: 1 g boiled with 100 ml of water.

Strontium chloride: **0.1 M**, 13 g of $SrCl_2 \cdot 6\ H_2O$.

Strontium nitrate: **0.5 M**, 106 g of $Sr(NO_3)_2$.

Sucrose (sugar): **0.1 M**, 34 g of $C_{12}H_{22}O_{11}$.

Sugar, dark brown: 5 g of dark brown sugar.

Sulfuric acid: 2.1, Add 670 ml of conc. sulfuric acid slowly and with stirring to 330 ml of water: **Dilute** (1:6), Add 145 ml of conc. sulfuric acid slowly

and with stirring to 870 ml of water; **Dilute** (1:11), Add 83 ml of conc. sulfuric acid slowly and with stirring to 917 ml of water.

Tartaric acid: **0.5 M**, 75 g of $H_2C_4H_4O_6$.

Tin(II) chloride: **0.1 M**, Dissolve 23 g of $SnCl_2 \cdot 2\ H_2O$ in 35 ml of conc. hydrochloric acid. Dilute to one liter with water. Add a few granules of tin. Should be freshly prepared.

Zinc chloride: **0.05 M**, 7 g of $ZnCl_2$.

Zinc nitrate: **0.1 M**, 30 g of $Zn(NO_3)_2 \cdot 6\ H_2O$.

Zinc sulfate: **0.5 M**, 144 g of $ZnSO_4 \cdot 7\ H_2O$; **0.25 M**, 72 g of $ZnSO_4 \cdot 7\ H_2O$; **0.2 M**, 58 g of $ZnSO_4 \cdot 7\ H_2O$.

For Exp. 60: Stock solution, each ml contains 10 mg of metallic ion, 46.5 g of $Cu(NO_3)_2 \cdot 6\ H_2O$, 53 g of $Mn(NO_3)_2 \cdot 6\ H_2O$, and 45.5 g of $Zn(NO_3)_2 \cdot 6\ H_2O$.

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